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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

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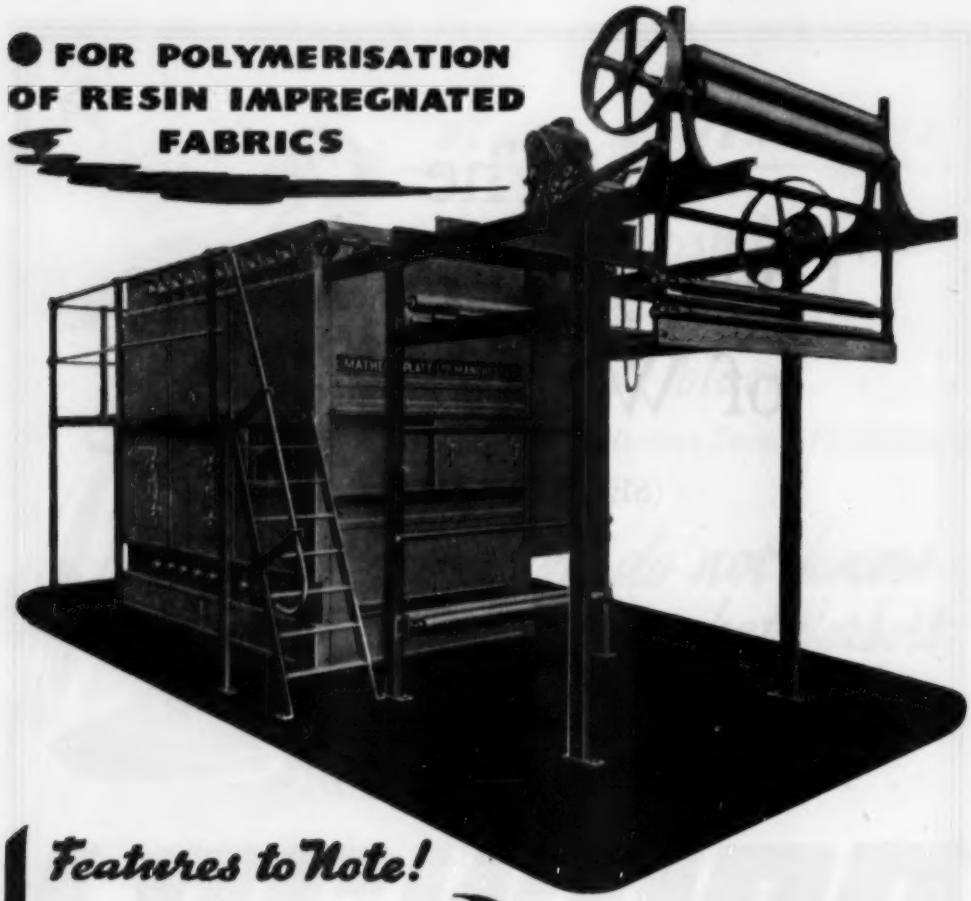

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1954 and pages 229–232 of the July 1953 issues of the *Journal*, or write to *The Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURE

Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons — *B. Kramrisch and A. E. Bratt*

COMMUNICATIONS

A Laboratory Skein-dyeing Machine — *W. Armfield*

The Polarography of Azo Dyes — *J. de O. Cabral and H. A. Turner*

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Proceedings of the Society

The Use of Organic Colouring Matters in Plastics

C. R. M. Oehlcke

Meetings of the West Riding Section held at Leeds University on 18th November 1952, Mr. S. Peel in the chair; of the Manchester Junior Branch held in Manchester College of Technology on 2nd March 1953, Mr. J. Boulton in the chair; and of the London Section held in the rooms of the Royal Society, Burlington House, Piccadilly W.1, on 2nd October 1953, Mr. G. F. Wood in the chair

The term "plastics" is now frequently used, and the past ten years have seen a rapid expansion in the number of types that are in regular use. Owing to these developments, the term "plastics" is about as useful as the word "textiles". Any dye manufacturer, if requested to supply a dye for textiles, would have to know the nature of the textile, and the same position is true in the plastics field to-day. Each plastic has to be considered separately, just as, for instance, no-one would make the mistake of thinking that a dye suitable for cotton would necessarily be suitable for wool.

Plastics can be divided into three main groups—

(1) Protein, such as material made from casein.

(2) Thermosetting, in which the material is first softened under heat-treatment, so that with pressure it can be moulded. Further heating, however, sets the plastic, which cannot be re-softened and remoulded. Under this heading come the well known phenol–formaldehyde, urea–formaldehyde, and melamine–formaldehyde resins.

(3) Thermoplastic—Such material can be moulded, and if heated again, softened, and can then be remoulded at the right temperature. Under this heading are—cellulose acetate, cellulose aceto-butyrate, cellulose nitrate, polymethyl methacrylate, polystyrene, polythene, and polyvinyl chloride.

This list, by no means exhaustive, will give some idea of the large field now covered by the term "plastics".

Some processing conditions are dealt with later, but at first some of the fastness requirements of the colouring materials may be mentioned. Owing to the wide range of articles made with various plastics, even after finding a satisfactory colorant to withstand the processing, the conditions of use of the article may impose further stringent requirements on the colouring matter. A short list of agents likely to be encountered is as follows—

(1) Acids (battery cases, container stoppers).

(2) Alkalis (toilet accessories, container stoppers).

(3) Solvents, oils, and greases.

The number of ways in which any article can meet such agents likely to abstract colour from the plastic article are too numerous to mention. A car component is always likely to meet oil, and toilet accessories may contain powerful solvents in the form of alcohols, ketones, and esters used in perfumes. Spirit soaps have a powerful penetrating and solvent effect.

(4) Hot water and steam. Any article used for medical requirements may have to withstand continual sterilisation, which is usually done with steam.

(5) Light. The requirements may vary between short-life articles such as toys, and long-life permanent household fittings.

The foregoing remarks indicate the wide nature of the plastics field, and in this paper the problems of colouring cellulose acetate, polyvinyl chloride, and polystyrene will be considered, but the principles and experience gained in colouring any one can be of guidance for others.

Classes of Organic Colouring Matters

The easiest way to colour any plastic is to use a soluble dye. By "soluble" is understood not necessarily solubility in water, but solubility in either the plastic or any plasticisers which might be added. These dyes are usually those soluble in either water, alcohols, esters, or hydrocarbons. Soluble dyes, however, often have serious drawbacks, and wherever possible colour in the form of a pigment is generally preferred. Surface dyeing of plastics is not often done, and generally it is the practice to colour the whole of the mass. Some of the water-soluble dyes can be made into insoluble lakes on a suitable base or extender. Such dyes are familiar to many in the dyeing trade under the heading of acid, direct, or basic dyes.

Another class of soluble dyes can be looked upon as half way between the soluble acid dyes and insoluble pigments; they are certain special lake-forming dyes which show a good degree of fastness

to solvents and light. These are well known in the trade under the names of Lake Red C (*C.I.* 165), Lithol Red (*C.I.* 189), Monolite or Lithol Rubines [1]. Such dyes also have the advantage that they can be precipitated without any extender, and the lakes are very often termed "toners". A third group of colouring matters for pigment application contain no lake-forming group and can be looked upon as true pigments. The range of such pigments is quite large, varying from those that are easily soluble in solvents to those that show good fastness to solvents.

A further group, which is really a subdivision of lakes, are products such as Alizarine Lake (Madder Lake, *C.I.* 1027), where the precipitating agent can be regarded as much more closely linked in the finished product and contributing to the colour of the lake. Such are the Alizarine and Helio Fast Rubine Lakes. Another main subgroup are the phthalocyanine pigments, which are again metal-complex compounds.

At present not many of the organic colouring matters used in the plastics trade are outside the above-mentioned categories.

Table I [2] summarises the tinctorial power of the various classes.

TABLE I

Colouring Matters	Tinctorial Power	Specific Gravity
Soluble dyes ...	High	1·2–1·5
Colour lakes ...	Medium	1·5–4
Toners ...	Medium to high	1·4–2·2
Pigments ...	High	1·2–2·2

The effect of specific gravity on the finished product is greater than the figures apparently indicate when they are considered with their tinctorial power, and this effect can be appreciable in the case of colour lakes.

A more detailed consideration of the various classes of colouring matters and their general properties is useful in assessing their possible use in plastics.

Soluble Dyes

Dyes soluble in esters, ketones, and hydrocarbons are on the market, for example under the names Sudan, Waxoline (ICI), and Zapon Fast. Some of the disperse dyes are also used. Their use is chiefly limited to the production of transparent articles and their fastness to light in this form is often quite good, but if the material is rendered opaque by the use of titanium dioxide, light fastness is usually poor. Further points to be considered in their use are the possibility of sublimation and liability to migrate in the material, when the dye can be rubbed off the surface of the article after it has been stored for some time.

Colour Lakes

These are usually barium precipitations of acid or direct dyes on an alumina–blanc fixe base. Generally they exhibit poor to moderate light fastness and the tinctorial value is low. Their resistance to plasticisers is good, but in view of their shortcomings their use is limited to special cases. Some of the anthraquinone dyes which require alumina as an essential part of the lake (e.g. alizarin lakes) show better fastness to light. Generally products from this class are useful where transparent to translucent results are desired.

Toners

This term is usually meant to cover lakes of dyes where the sulphonic acid group does not exert sufficient effect on the dye to render it completely soluble in water. Their use, therefore, is confined to precipitating them completely as lakes usually containing no added base. Their tinctorial value is high and fastness to plasticisers very good. The light fastness may be good, though generally in pale shades it is poor, so that care is needed in their use.

Pigments

This term is meant to cover colouring matters that contain no solubilising groups. They are nearly all azo pigments, and these range from yellow to maroon. Many are used in the printing ink and paint trades, some of them having very good light fastness, even as tints, but unfortunately, a great number bleed badly in plasticisers, which rules out their use in many cases. Some of the best pigments for printing inks are completely ruled out on account of poor fastness to solvents and plasticisers. The more modern brands of the azo range contain a benzidine or substituted benzidine tetrazo component with acetoacetarylamides and pyrazolone derivatives and show more resistance to the action of plasticisers. In view of the exacting requirements of a colouring matter for use in polyvinyl chloride, it is perhaps too early to say that they can safely be used for this purpose.

Other pigments outside the azo class are the phthalocyanines, Pigment Green B (iron derivative of α-nitroso-β-naphthol), and anthraquinone and indigoid vat dyes. These generally are of good fastness to light and plasticisers.

GENERAL PROPERTIES OF PIGMENTS

There are certain characteristics of pigments which have to be taken into consideration when using them as standards in the works. Many pigments are *dichromatic*, which means that the hue will vary with concentration, or in the case of transparent media, with thickness of the film. Many reds, for example, exhibit a bluish undertone, and very often when such a colouring matter is used for shadings, it is the undertone that is more effective than the full colour.

A number of pigments have a tendency to bronze, and this effect is evident when they are used at high concentrations. Other physical characteristics, such as bleeding or solubility in plasticisers, are important, and these properties will vary according to the nature of the plastic. The great importance of these characteristics will be seen, for example, in the colouring of polyvinyl chloride.

Dispersion

The state of dispersion and the particle size of a pigment or lake are important. Particle size should be such that practically 100% of the material will pass a 200-mesh sieve. Even when this is achieved, it may be that the pigment is not easily wetted by the plasticiser; in such cases the

flushing of a pigment into the plasticiser ensures better results, or the grinding of the dry pigment into the plasticiser, leaving the resultant paste a few weeks before finally milling on a three-roll printing-ink type of mill.

Melting Point

Though the melting points of the pigments are above processing temperatures, certain pigments tend to soften and cake on the rollers whilst being milled into the plastic. If this occurs, it is very often difficult to remove such specks of colour from the surface of the roll. In such cases, previous grinding of the colouring matter in plasticiser overcomes the trouble.

Light Fastness

It must be remembered that very few organic pigments show good light fastness when used as tints. The phthalocyanine group are exceptional in this respect. Very few reds, yellows, and oranges combine good fastness to light in tints with the requisite fastness to plasticisers, and it is the combination of these two factors which in many cases renders the choice of a suitable pigment difficult. Lack of appreciation of this point is likely to lead to many complaints. Many reds show quite good fastness when used for producing a red, but are very poor when used for a pink. This has made many manufacturers use only cadmium reds, so as to be always within safe limits. Owing to the high price of these latter products, their use can prove unnecessarily expensive for deep shades and cheap articles.

Users in the textile trade have long exercised care when making blends of dyes to produce a mixture dyeing. The need for this is just as great in the plastics field, and perhaps the difficulties are greater. For instance, when making a three-colour blend, if one of the components is present in only a small amount, its light fastness will be its fastness when used as a tint, the other two components apparently exercising no protecting influence. On exposure, the change in tone might be quite rapid, and this is clearly shown when using an organic colouring matter to shade an inorganic pigment.

A further fact to bear in mind is that the standard required is governed by the use and the life of the finished article and also that the fastness of a pigment varies in different plastics. The great difference in fastness is well illustrated by taking the results of some tests that were made in vinyl chloride copolymer resin (Table II); the discrepancy in this case between the light fastness of the various pigments and the fastness usually associated with them in printing inks and textiles is clearly seen.

These light tests were done with a carbon arc light source.

The foregoing results are surprising when the fastness to light usual with vat dyes on textiles is considered. Whilst the writer has not made duplicate tests, other tests have not rated the vat dyes as low as the above table. It may be that not only the action of light, but the quality of polyvinyl chloride, played a part in the results. The table, however, does clearly show the necessity for careful testing without preconceived ideas gained from other fields.

TABLE II

Fastness to Light of Pigments in Vinyl Chloride–Acetate Copolymer Resin[3]

Pigment	Light Fastness*
YELLOWS	
Benzidine Yellow	C–D
Hansa Yellow	C–D
ORANGES	
Benzidine Orange	C
Nitraniline Oranges	D
REDS	
Madder Lake	D
Hansa Rubine G	B
Lithol Reds	D
Lithol Rubines	B
Indanthren Pinks	E
Lake Red C	C
Thioindigos	E
Toluidine toners	D
GREEN	
Phthalocyanine Green ...	A
BLUES	
Phthalocyanine Blues ...	A
Indanthren Blues	C
Victoria Blue (phosphotungstic acid lake)	C

* A = Excellent E = Poor

The benzidine pigments mentioned in the table are organic pigments obtained by coupling tetrazotised benzidine derivatives with, for example, acetoacetanilide or 3-methyl-1-phenyl-5-pyrazolone.

FASTNESS REQUIREMENTS

Naturally every manufacturer would like to have every shade of colour of the highest possible fastness at the lowest price. As is well known in the case of textiles, this wish cannot be realised, and possibly, if between suppliers and users of colours more information were exchanged as to the ultimate use of any colour, a colouring matter of the requisite fastness could be found, cheaper in price than using the most expensive and fastest for the job.

At one time the polyvinyl chloride trade used a number of colouring matters which had limitations as regards light fastness. Certain articles showed up this factor, and cadmium pigments, for example, were preferred for all purposes. A few years ago the price of the finished article did not matter, so that high cost of colouring passed, perhaps, unobserved. To-day, however, the position has changed, and where a finished article has only a short life, prices are becoming keener. It is found unnecessary in many cases to go to the expense of colouring with the fastest possible colour. The position is like the dyer deciding in some cases to use a direct dye, and in others a vat dye, for producing the same colour but for a different class of article in each case. Unfortunately for the plastics manufacturer, it is not possible to classify the fastness of organic pigments as definitely as with textile dyes. The classification depends quite as much on the article that is to be made as on

the colouring matter in it, and any final assessment of fastness has to be on the particular article produced. It is now becoming the practice more and more to use the light fastness scale 1–8, which is the standard in the textile trade [4].

THE COLOURING OF CELLULOSE ACETATE

The colouring of cellulose acetate will illustrate the application of some of the foregoing principles and can be taken as representative of the group of cellulose plastics. Cellulose acetate is widely used for injection moulding, and a wide range of articles are made with it. The average material usually contains about two-thirds polymer and one-third plasticiser such as diethyl and dimethyl phthalate. The behaviour of any colouring matter towards the plasticiser is, therefore, of importance. In this instance the chief factors to be taken into account are—

(1) Action of heat

(2) Action of plasticiser

(3) Opacity of finished product

The usual method of colouring the acetate is to soften the material on a mill with the plasticisers, following by the addition of fillers. On cooling, the mass sets hard and can then be ground to chip form suitable for moulding. The temperatures for injection mouldings are in the region of 190°C., and at this temperature some solubilising effect on the colouring matter is sure to occur. To ensure even colouring with a soluble dye, presolution of the dye in a suitable solvent, such as acetone or alcohol, is necessary, the small quantity necessary being lost during the time of milling. For transparent effects, soluble dyes are nearly always the best, and even dyes which are normally regarded as of poor fastness show quite good fastness when used for this purpose. A probable explanation is that fading does occur in the first few layers of the material, but is masked owing to the transparency of the material, allowing the colour of the lower layers to be seen. The difference in apparent fading between a thick and thin sheet seems to confirm this. When the material is rendered opaque with a pigment such as titanium dioxide, the fastness of the colour is very much lower. Some soluble dyes show quite good fastness (Zapon Fast dyes). With all soluble dyes, however, there is always likely to be the defect of the colour slowly migrating to the surface of the plastic, with the resultant lack of fastness to rubbing. This can occur even at very low percentages, when it might be expected that there is sufficient cellulose acetate to hold any dye. For most work, therefore, a colour in pigment form, insoluble in plasticisers, is to be preferred.

Whilst the best transparent effects are obtained with soluble dyes, it is possible to obtain good transparency by the use of transparent lakes, especially if these are first dispersed in the plasticiser. Such colourings show no migration effect, and therefore no rubbing off when the article is aged. For opaque shades, toners and pigments are used. A general list of colouring matters is as follows—

SOLUBLE

Certain acid dyes
Zapon Fast, Grasol, Resoform, Resin, etc.
Sudan

INSOLUBLE

Benzidine Orange, Benzidine Yellow
Helio Fast Red RL (Monolite Fast Scarlet)
Phthalocyanine pigments
Lake Red C (barium lake)
Lakes of soluble anthraquinone dyes
Anthraquinone vat dyes.

THE COLOURING OF POLYVINYL CHLORIDE

This is one of the most difficult materials to colour satisfactorily with organic colouring materials. The difficulties were not appreciated in the early years, and factors likely to influence the choice of colouring matter were not fully known. At first, pigments generally used for rubber were recommended, and superficially it seemed at the time that polyvinyl chloride was an easier colour problem than rubber, as limitations such as freedom from copper and manganese and fastness to open steam cure did not arise with polyvinyl chloride plastics. These drawbacks, however, are replaced by others, making the choice of suitable pigments difficult.

There are two main factors affecting suitability of colouring matters, one is the polymer and the other the added ingredients, the chief of which is the nature of the plasticiser; before going into detail, the processing conditions can be noted. The polymer in the form of a fine white powder is mixed with the necessary pigments, plasticiser, and stabiliser in a mixer of the Z-blade type. The resulting mix, which is in the form of a crumb, is then milled for a short time (about 6 min.) in a "Banbury" at about 120–130°C. This is followed by a more intimate mix on a rubber mill; at this stage little gelling has taken place. Finally, calendering or extruding is done and the temperature is about 160°C. or even higher. Whatever the variations in manipulation, a temperature of 160°C. is reached at some time, and perhaps even higher for a few seconds, as, for example, in an extrusion head. The polymer under such conditions gives off some hydrochloric acid, and to prevent the deteriorating effect on the polyvinyl chloride, a stabiliser such as lead carbonate is added. If, however, there are traces of a peroxide left in the material (used as polymerisation catalyst), it is quite likely that chlorine is liberated, or at any rate, conditions of acid oxidation are set up. Owing to the improved quality of the polymer supplied to-day, this severe condition is not likely to be met, but in the early years there were grades of polyvinyl chloride whose effect on colouring matters was explained on these lines; for instance, dyes of the phthalocyanine group, normally of excellent fastness, were easily discoloured or even totally bleached. Other colouring matters, such as Lake Red C (barium lake), Permanent Red 2B barium lake, and Benzidine Orange, were greatly changed in colour, and the resulting coloured compound was usually of much lower fastness to light than normal. Whilst such an effect is easily seen in self shades, it may pass unnoticed when a mixed shade is made; the poorer light fastness will

be discovered later, perhaps when the finished material reaches the customer. In general, it is not so much the heat that affects the colour. The range of dyes of good heat fastness, taking heat alone into consideration, is quite large, but it is what happens in the compound at any particular temperature that is important. Before considering the colouring of polyvinyl chloride, a review of the behaviour of the main types of colouring matters in the plasticisers is useful and instructive.

Table III is a condensation of about 300 results, from which the following conclusions can be drawn—

1. Dibutyl phthalate has a greater solubilising effect than dioctyl sebacate.
2. Dibutyl phthalate generally is more severe in action than dioctyl phthalate and tricresyl phosphate, though occasionally, as for instance with alizarin lakes, tricresyl phosphate has a stronger effect.
3. Many pigments darken temporarily under the conditions of the test but return to their original colour on cooling.
4. Certain pigments tend to agglomerate.
5. Phosphomolybdotungstic acid lakes of basic dyes go sticky and are apparently completely decomposed. Their fastness becomes the fastness of the basic dye and their good fastness to light in printing inks is not shown in many plastics.
6. Certain pigments become soluble at 180°C., but are practically insoluble cold. Such pigments might be expected to crystallise from the plastic mass and perhaps cause a colour deposit which can be rubbed off the surface of the article.

From Table III can be surmised what might happen to a colouring matter. Where a colour bleeds appreciably in the plasticiser, it is nearly certain that trouble can be expected. The plasticiser is slowly lost by the finished material, so that a polyvinyl chloride sheet cannot be looked upon as static. There is always movement of plasticiser to the surface, where it is lost by evaporation. Any pigment, even in very small percentages, dissolved in the plasticiser will be carried to the surface and there deposited. This phenomenon, known as "migration", is one of the chief difficulties in finding suitable organic colouring matters for polyvinyl chloride. A standard test [5] is laid down in which coloured sheet is placed between white sheet for a period of 72 hr. at 50°C. under a pressure of 3 lb./sq.in. This test will normally pick out any colour with a tendency to migration. However, some colouring matters do not always behave the same: pigments of the Benzidine Yellow type, in some

TABLE III

Tests in Plasticisers at 180°C. and Cold Styrene Monomer*

	D.O.S.	D.B.P.	B.A.R.	T.C.P.	D.O.P.	Styrene	Remarks
Lake Red C barium	v.s.B.	B.	N.B.	v.s.B.	s.B.	v.s.B.	Temporary darkening
Lithol Red R barium	B.	S.B.	N.B.	S.B.	S.B.	N.B.	Permanent darkening
Lithol Red R calcium	N.B.	v.s.B.	N.B.	s.B.	v.s.B.	N.B.	Temporary darkening
Permanent Red B barium	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	Slight darkening
Permanent Red 2B barium ...	N.B.	s.B.	N.B.	s.B.	v.s.B.	N.B.	Slight thickening, temporary darkening
Permanent Red Toner NCR (FH)	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Permanent Red Toner 4B calcium ...	N.B.	N.B.	N.B.	N.B.	v.s.B.	N.B.	Temporary darkening
Pigment Claret BBN (calcium) (Newcol)	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	Slight temporary darkening
Helio Fast Red RL	sol.	sol.	sol.	sol.	sol.	sol.	
Permanent Red FRL (FH) ...	sol.	sol.	sol.	sol.	sol.	sol.	Comes out on cooling
Helio Fast Pink RL (FBy)	sol.	sol.	sol.	sol.	sol.	sol.	Change of hue to orange
Indanthren Blue GGSL (Basf) ...	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Fanal Blue B (Basf)	v.s.B.	s.B.	s.B.	s.B.	s.B.	N.B.	
Fanal Yellow Green G Supra Powder (Basf)	B.	S.B.	N.B.	Sol.	S.B.	B.	Sticky
Permanent Carmine FB (FH) ...	s.B.	B.	s.B.	S.B.	v.s.B.	v.s.B.	
Vulcan Fast Red B (FH)	N.B.	s.B.	N.B.	B.	s.B.	v.s.B.	
Helio Fast Rubine 3BL Lake (FBy) ...	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Pigment Blue RT (Newcol) ...	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Madder Lake 5133 (Newcol) ...	N.B.	N.B.	N.B.	S.B.	N.B.	N.B.	Darkens at 180°C. Remains duller
Madder Orange Lake 5134 (Newcol)	N.B.	v.s.B.	N.B.	sol.	N.B.	N.B.	
Pigment Brown R 5364 (Newcol) ...	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Pigment Green B	N.B.	N.B.	N.B.	s.B.	s.B.	N.B.	Slight dirty bleed may be impurity
Monastral Fast Blue BS Powder (ICI)	N.B.	N.B.	N.B.	N.B.	s.B.	N.B.	
Monastral Fast Green GS Powder (ICI)	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	
Benzidine Orange	B.	S.B.	s.B.	B.	B.	S.B.	Agglomeration occurs Increase in opacity
Pigment Yellow GRR Extra (Newcol)	B.	S.B.	B.	sol.	S.B.	S.B.	
Benzidine Yellow	s.B.	s.B.	v.s.B.	B.	s.B.	s.B.	Coagulates

*D.O.S. = Dioctyl sebacate
D.B.P. = Dibutyl phthalate
B.A.R. = Butyl acetylricinoleate
T.C.P. = Tricresyl phosphate
D.O.P. = Dioctyl phthalate

B. = Bleeds
N.B. = Non-bleeding
v.s.B. = Very slight bleed
S.B. = Strong bleed
s.B. = Slight bleed

compounds, will give a perfectly satisfactory result; the same pigments when used with a blue to make a green will migrate. Generally it has been the writer's experience that mixtures of the pigments behave worse than the single components. It may be that the susceptible colouring matters become more widely and finely dispersed over the surface of the other ingredient and so offer a greater surface area to attack.

The other chief factor in determining the choice of colouring matter is that of light fastness. Here the same remarks about the degree of opacity or transparency of the material made about cellulose acetate apply. In the case of opaque materials where titanium dioxide is usually used as the white pigment, the fastness is the lower the paler the shade. It is in the case of pale shades that the lack of really fast organic pigments is felt. For pale reds and yellows, the cadmium pigments are still generally preferred, their drawback, however, being "deadness" of tone when used below a certain strength, and this is particularly evident when transparent colourings are made.

As much coloured polyvinyl chloride is used in the electrical industry, the effect of any pigment on the insulating resistance of the material has to be considered. Complete washing of the pigment after manufacture so as to remove any soluble electrolytes is essential, but even when this has been done there is always a residual effect due to the chemical nature of the pigment. Pigments containing sulphonic acid groups tend to lower the resistivity of the compound. This effect is particularly shown in the choice of suitable reds for cable work. Polar groups increase resistance to migration but are bad electrically. On the other hand, there are many red pigments containing no such groups, but unfortunately they tend to be soluble in solvents and plasticisers. A compromise, therefore, has usually to be made, by, for example, using a pigment having good electrical properties but perhaps lower light or plasticiser fastness. The choice can be made only where the exact nature of the articles being made is known and any latitude in fastness properties in any direction can be assessed.

THE COLOURING OF POLYSTYRENE

The colouring of polystyrene is to some extent made simpler by the fact that no plasticiser is needed. The material flows quite freely at about 200°C., and it can be softened and milled below that temperature. The resin can be coloured on a mill or by merely mixing the colouring matter with the resin and feeding immediately to the injection moulder. This latter technique requires that the polystyrene is in a suitable form and that it is pretreated with an agent to ensure the even spreading of any white pigment and colouring matter over the surface of the resin [6].

The colouring matters can either be pigments or soluble dyes, solubility in this instance referring to solubility in polystyrene. Certain oil-soluble dyes, marketed under the names, "oil-soluble", "Waxoline", "Sudan", etc., have been found satisfactory; their chief use is, however, for transparent articles.

In the dry powder mixing technique, it is important that the following two conditions are fulfilled—

(1) The fine division of the colouring matter. This ensures even and wide distribution of the colour over the resin particles.

(2) Rapid solution of the colour in the case of soluble dyes.

An investigation carried out in the writer's laboratory showed that dyes freely soluble in the monomer gave different results in injection moulding by the dry powder method. For instance, differences between Sudan I (*C.I.* 24) and Sudan II (*C.I.* 73) were found, the former dissolving much more rapidly than the latter.

When these dyes are used for transparent articles, the fastness to light is generally adequate, for the same reasons that fugitive dyes show good fastness to light in the other transparent plastics. If, however, they are used to make an opaque moulding by incorporating a small amount of titanium dioxide, the fastness is poor. For this reason, for opaque shades, pigments are to be preferred. It has been the writer's experience that, even though a pigment may show greater or lesser solubility in styrene, when used for the colouring of polystyrene, the finished article does not show any tendency of the colour to migrate or bloom. There can be no movement of plasticiser as in the case of other plastics. This fact allows a wider choice of pigments than is the case with polyvinyl chloride. There is one possibility, however, of trouble: certain pigments soluble in polystyrene, when moulded, show a different colour from that normally associated with them. Certain reds in polystyrene are yellower in tone than when they are, for example, used for a printing ink. This is due to the colour of their solution being different from that of the pigment. Besides making colour matching difficult, if such products are used it might be that after a period of time, crystallisation of the pigment will develop with a resultant change in colour of the article. Tests are not yet complete to verify this, but this phenomenon has been encountered in the case of cellulose acetate mouldings.

CONCLUSION

In order to obtain the highest possible fastness we have to look more and more towards pigments. The weakness of pigments up to the present has been their lack of fastness to solvents and plasticisers. There are three ways of making colouring matters non-bleeding in solvents—

(1) Introduction of strongly acid groups such as sulphonic. These, however, mean that a lake has to be made, with consequent loss in tinctorial power.

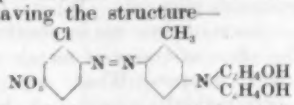
(2) By introducing weakly acidic radicals, either non-laking such as nitro or weakly laking such as carboxyl.

(3) By increasing the size of the molecule.

These results, especially (2) and (3), can be seen in the following colouring matters of fairly recent introduction.

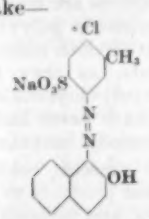
A consideration first of the types of molecule soluble in solvents emphasises the totally different types from those which tend to be insoluble.

Simple dyes containing no lake-forming groups, of the type Sudan Orange G (aniline→β-naphthol), Helio Fast Red RL (*m*-nitro-*p*-toluidine→β-naphthol), Sudan Red (amino-azotoluene→β-naphthol), Dispersol Fast Yellow G (*p*-nitroaniline→aniline), are all freely soluble in solvents, though insoluble in water. Developments of these types are shown in Celliton Fast Rubine 3B (000) having the structure—



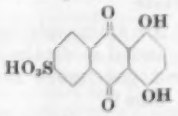
and Sudan Red BV, where one C_2H_4OH is replaced by butyl C_4H_9.

Very much better fastness to solvents is obtained by introducing sulpho groups, as in dyes like Lake Red C, which is usually marketed as the concentrated barium lake—



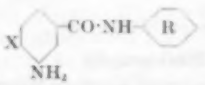
If the methyl group is oxidised to carboxyl, little change in colour occurs, but better solvent fastness than Lake Red C is shown. The Permanent Red range of lakes are made by using 3:2-hydroxynaphthoic acid instead of β-naphthol as coupling component.

The lakes of Alizarin Red and Orange show good fastness to solvents and plasticisers, but both show lack of stability to tricresyl phosphate. The effect of introducing sulphonic acid groups is to give much greater stability, as is shown by the lakes of the Helio Fast Rubine series. Helio Fast Rubine 3BL (FBy), for example, has the structure—

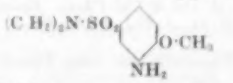


Simple aniline derivatives coupled with acetoacetarylides have poor fastness to solvents and plasticisers. If, however, benzidine derivatives are used, much better fastness is obtained. These products were first marketed under the name Vulcan Fast Yellow, the GR brand, for example, having the coupling dichlorobenzidine→acetoaceto-*m*-xylidide[1]. A further development was to use Naphtol AS–G as a coupling component with monoamines. These only produced yellows and yellowish oranges. Oranges and reds were obtained by using methylphenylpyrazolone and its derivatives as a coupling component with benzidine derivatives for the amine. The first of these colouring matters was Permanent Orange G (FH) (Monolite Fast Orange GS) (ICI) having the coupling dichlorobenzidine→methylphenylpyrazolone.

The earlier couplings of the Naphtol AS type, whilst giving some pigments of very good light fastness, unfortunately showed very poor fastness to solvents and plasticisers. Amines, however, of the type—



give pigments of good fastness to solvents, an example being the product that was marketed under the name Vulcan Fast Rubine B (FH)[1]. Another useful amine is of the type—



An example of this type was marketed under the name Permanent Carmine FB (FH) having the coupling Fast Red ITR Base→Naphtol AS–ITR[1].

Pigments of the phthalocyanine series such as Monastral Fast Blue BS (ICI) show excellent fastness, the range at present, however, only covering mid blues to bluish greens. Certain anthraquinone vat dyes show promise, but many have the drawback of being to some extent soluble in plasticisers, and also their development has no doubt been held back by their high price and lower tinctorial value than pigments of similar colour in other chemical classes.

Up to quite recently the colouring of plastics has been a rather hit-or-miss method, but perhaps the results illustrated do show that it is possible, knowing what are the requirements, to start looking in the right class for a colouring matter to fulfil a certain specification.

Plastics are still in their infancy, and the coming years will no doubt show a great expansion. This will bring its problems, but these are to be welcomed as they are ever the stimulus to greater efforts to find new and better products.

We have not yet learned properly how to make the most of plastics, and they are still often looked upon as substitute materials. This will change, as they have their characteristic properties, assets, and defects. The type of colouring matter available and the influence of molecular size, groups, and their effects on their physical properties have been indicated.

The polymer manufacturers make polymers to suit a particular purpose, and colour manufacturers similarly make colouring matters for a particular purpose; for steady progress to be made, close co-operation between the colour user and manufacturer is essential, as conditions are changing so rapidly. With such co-operation the gaps in the range of suitable colouring matters for various purposes will slowly be filled to the advantage of both colour user and manufacturer.

* * *

Many of the points discussed in this paper were illustrated by colorations of the various plastics.

NEWCOL LTD.
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BRINKSWAY
STOCKPORT

(*MS. received 26th November 1953*)

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Discussion

WEST RIDING SECTION

Mr. E. R. INMAN: The lecturer mentioned the fading of a red shading colouring matter. Would he say that it is a general rule, in the colouring of plastics, that the components of a mixture fade at rates which are independent of one another?

Mr. OEHLCKE: From personal experience the use of a fugitive colouring matter produces poor fastness and the components do seem to fade at rates independent of one another and more in proportion to the actual percentage present in the compound.

Mr. INMAN: The lecturer has studied the constitution of colouring matters from the point of view of fastness to bleeding and he observed that anthraquinone vat dyes showed indifferent fastness to light. Has any study been made of the constitution of vat dyes with the object of obtaining some guide as regards fastness to light? For example, Monastral Blue exhibits very good fastness to light in contrast to Anthraquinone Blue, and it may be that some of the vat dyes of molecular size intermediate between those two products will exhibit very good light fastness.

Mr. OEHLCKE: A detailed study of the vat dyes has not been made, as, owing to their high price and comparatively poor staining value, an investigation did not seem worth while as against the study of colouring matters of similar colour, e.g. in the azo classes. Poor results in fastness to light have been obtained, if not quite so bad as those indicated in Table II.

Mr. E. E. SUNDERLAND: Will the lecturer differentiate between the two methods of pigmenting polyvinyl chloride, viz. (i) milling the pigment into the polyvinyl chloride–plasticiser mix, and (ii) mixing the pigment with the polyvinyl chloride prior to adding the plasticiser, with reference to the ease of the operation and the properties of the resultant final material?

Mr. OEHLCKE: Where a pigment has a soft texture and is finely ground, it can be milled easily either into the polyvinyl chloride–plasticiser mix, or by adding it with the dry polymer and then adding the other ingredients. If, however, the pigment has a hard texture, it is not possible to obtain full development of the colour in the plasticised compound, and this applies particularly to lakes in which alumina is present. In such cases the best results and tinctorial value are obtained by dispersing and developing the colour in the plasticiser before adding to the vinyl chloride polymer.

Mr. L. YARKER: Has the lecturer tried—

(1) Colouring matters with non-migratory plasticisers, e.g. polypropylene adipate?

(2) Iron oxide pigments— What is the lecturer's opinion of their use in plastics?

(3) The effect of traces of metals such as zinc and copper— What are the limits permissible?

Mr. OEHLCKE: (1) No comparative tests have been made with non-migratory plasticisers, and such tests, if carried out, should prove informative.

(2) Diverse opinions are met with on the value of iron oxide pigments in polyvinyl chloride. For example, some leathercloth manufacturers will not use these pigments, as they have experienced degradation of the polyvinyl chloride with their use. Others, however, have never had any trouble and have used them happily for years.

(3) Up to the present, colour manufacturers have not had to test products to permissible limits of metal impurities, and no tests are laid down as, for example, in the rubber trade. Zinc is well known to catalyse the decomposition of polyvinyl chloride, and it may be that even traces will show this effect. No reports seem to have been published showing that traces of copper have a bad effect.

Mr. R. K. FOURNESS: What range of percentage of colouring matter is used?

Mr. OEHLCKE: The percentage of colouring matter needed depends to a large extent on the amount of covering white in the article being made, and additions will vary between about 0·02% for transparent articles to 2% for an article loaded with titanium white.

Mr. FOURNESS: Are aminoanthraquinone acetate-rayon dyes used, and if so do they show the same weakness to burnt gas fumes as when dyed on cellulose acetate fibre?

Mr. OEHLCKE: The author has not met cases of the "gas" effect with dyes of the aminoanthraquinone acetate rayon classes; it may be that the reason for this is that the plastic gives sufficient protection to the colouring matter.

Mr. E. THORNTON: Should colouring matters (not of the lake type or containing sulphonic acid groups) have melting points higher than the maximum heat-treatment temperature encountered in processing?

Mr. OEHLCKE: High melting point is an advantage, as dyes whose melting point is not far from processing temperatures usually bleed or migrate in plastics, the exception, of course, being the colouring of polystyrene. If also the pigment contains impurities, it may tend to stick to the milling rolls and be very difficult to remove from them into the compound that is being worked.

MANCHESTER JUNIOR BRANCH

Mr. W. W. SWORD: Do the dyes used for colouring plastics change hue to any degree according to the plastic on which they are used?

Mr. OEHLCKE: Providing the dye withstands the processing of the particular plastic there is not much variation in the colouring produced, though different plastics vary in appearance, which reflects itself in the degree of brightness.

Mr. SWORD: Is there a range of fluorescent dyes suitable for colouring plastics, and if so, do they have the disadvantage of possessing poor light fastness, as applies usually with textiles?

Mr. OEHLCKE: Up to the present there has been no demand for fluorescent dyes for plastics. Fluorescent effects can easily be obtained with dyes such as Rhodamine, which, however, has the disadvantage of poor fastness to light except in transparent colourings. It is to be expected that fluorescent pigments at present on the market would show the same poor light fastness in plastics as is associated with their use in the printing ink field.

Mr. C. SHRIVE: What is the effect of the plasticiser on the light fastness of pigments?

Mr. OEHLCKE: It has not been noticed that the plasticiser affects the light fastness of pigments in plastics. Where the plasticiser tends to turn yellow on exposure to light, this effect may be pronounced in pale colourings.

Mr. H. J. TWITCHETT: Is it really the phthalocyanine pigment rather than the sulphonated phthalocyanine lake which is decomposed in poor-quality polyvinyl chloride?

Mr. OEHLCKE: In the patterns which showed the effect of poor-quality polyvinyl chloride on phthalocyanine pigments, it was found in this particular instance that the sulphonated product showed better fastness.

Mr. H. G. WHITE: Which vat violet was referred to as giving anomalous behaviour on blooming?

Mr. OEHLCKE: Slight bleeding was encountered with Indanthren Red Violet RH when used as a shading component.

Mr. WHITE: Could the lecturer explain the only moderate light fastness of Indanthren Blue? My own work rated it at 7+ on the S.D.C. Scale (*B.S.* 1006).

Mr. OEHLCKE: The author's own experience is that Indanthren Blue GGS does give varying results under different conditions, though no personal results could be rated as low as that of Table II. It has to be borne in mind that blues are very much affected by any change in colour of the medium used, and this is very difficult to allow for when assessing results, particularly so in pale colourings.

Mr. K. W. JONES: Can Mr. Oehlcke say roughly what is the comparative use of organic against inorganic pigments, and what factors largely determine the choice of the former?

Mr. OEHLCKE: The great advantage of organic pigments is their brightness of colour and the small additions necessary, so that the physical properties of the plastic are not appreciably altered with their use. As has been indicated in the lecture, it is not always possible to obtain the necessary fastness, so that inorganic pigments still have their uses. In the case of browns, the low price of iron oxides is an additional attraction.

COMMUNICATION

Analysis of Textile Auxiliary Products*

J. A. VAN DER HOEVE

Since the previous paper[1] not only have a great many new products been marketed, but new groups of products have also been developed, such as the cation–anion-active compounds and the polyamides (not proteins). Both these new groups behave in many respects like polyethylene oxide compounds, and consequently are included according to the old system among the aminopolyethylene oxides. It has proved desirable to subject this latter group to further subdivision. In addition to this, even before the previous paper a number of groups were known which at the time, owing to lack of samples, had not yet been included in the analytical system, e.g. phosphonium compounds, alkylnaphthalenephosphonates, alkylamidoalkyl phosphates, alkylamidoalkanecarboxylates. The separation of the cation-active amines can now be continued much further than was possible at that time. These factors have induced me to reconsider the analysis of textile auxiliary products, and to attempt to classify them into a larger number of groups by means of the initial tests.

Introduction

The number of textile auxiliary products on the market is enormous and is constantly increasing. Identification of these products is itself a fairly exacting task, but this task can be enormously aggravated if mixtures of auxiliary products are to be dealt with. As in the previous paper[1], it is intended to discuss the identification of single auxiliary products, while at the end a few suggestions will be made for the examination of mixtures. It is essential to point out that some of the reactions may be interfered with by the presence of inorganic salts. If such interference is to be avoided, it is necessary to dry the auxiliary product at a low temperature and extract it with ethanol or methanol.

Discussion of the Literature

Of recent years two very important papers[2,3] on the analysis of auxiliary products have been published. Wurzschmitt[2], in addition to an analytical system for single compounds, also gives an analytical method for mixtures of auxiliary products. The scheme for single compounds is clear enough *per se*, but with the three tannin tests

* Synthetic detergents, wetting agents, restraining agents, and emulsifying agents.

there discussed I did not obtain the results to be expected. In particular, sulphated polyethylene oxide compounds and polyamines gave rise to discrepancies. The cation and anion tests yielded good results, as did also the two heating tests and the various iodine tests. The three iodine tests given by Wurzschmitt may be considered welcome additions to the KI_3 test. For the cation-active compounds the permanganate test may be looked upon as an asset. It has, however, been found during the testing with 55 cation-active compounds that with a number of quaternary ammonium compounds the chloroform layer is not coloured violet, but brown.

In the main scheme for mixtures, Wurzschmitt distinguishes 14 groups, depending on 14 reaction effects obtained with 8 reagents. As regards these reaction effects, those of the anion test and the cation test are the most important. It is to be regretted that it is precisely these tests which lead to different results from those described by Wurzschmitt. As a matter of fact, polyethylene oxide compounds greatly interfere with these reactions. During the testing of the influence of Iguafen O500 on the reaction of Solidogen BSE and Nekal BX it was found that no turbidity occurred at all. In order to obtain a better result, Nekal BX was replaced by Heliogen Blue SBL, which according to Wurzschmitt is one of the most powerful reagents for cation-active substances. However, this test also proved unsuccessful. The mixture of Iguafen O500 and Solidogen BSE (1 : 1) did not give any precipitate or turbidity with Heliogen Blue SBL. The Blancotan B test for anion-active substances was found less sensitive to admixtures of polyethylene oxide derivatives, but this reaction was interfered with just the same.

In addition to the analysis, Wurzschmitt also deals with the classification of textile auxiliary products. He takes quite a different view of polyethylene oxide derivatives from the prevalent one. He points out the numerous points of resemblance between the behaviour of cation-active compounds and polyethylene oxide derivatives towards various reagents. He concludes from this that the polyethylene oxide derivatives should be looked upon as oxonium compounds.

The turbidity in aqueous solutions of polyethylene oxide compounds is considered by Wurzschmitt[4] to be caused by the conversion of oxonium groups to normal ether bridges[4]. On the basis of measurements of the conductivity of such solutions above and below the turbidity temperature, I contested this view [5]. Indeed, in this temperature range the conductivity does not exhibit any abnormality, but rises regularly with the temperature.

Bergeron *et al.*[3] discuss the analysis of anion-active auxiliary products. Just as with Linsenmeyer[6], the main separation is based on three successive acid treatments of increasing intensity. The drawback that the alkylnaphthalenesulphonates behave very differently with regard to the boiling with acid also holds in this case.

During the discussion of the reactions to be included in the following analytical scheme, some of Wurzschmitt's and Bergeron's tests will be reverted to.

Analytical Scheme

In contrast with the previously published scheme, an attempt has been made in the main table to classify the auxiliaries into as many groups as possible. Further, boiling with sulphuric acid, which repeatedly tends to give rise to complications, has been minimised.

The main scheme is based on detection of any organically combined nitrogen that may be present, determination of the ionic nature of the compound (cation-active, non-ionic, or anion-active), and determination of the type of hydrocarbon group in the compound.

To this end the following tests have been used—heating with alkali (1), followed by the alkaline bromine test (2); cation (5) and anion (6) tests; the diazo test (7); Guerbet's test (8); and the starch–iodine tests (9).

The resulting classification of auxiliary products into thirty-six groups is set out symbolically in Table I.

TABLE I

Classification of Auxiliary Products

A ... No organically bound nitrogen present
B ... Organically bound nitrogen present
I ... Cation-active
II ... Non-ionic
III ... Anion-active
N ... Positive diazo test (7) (naphthalene nucleus)
P ... Negative diazo test and positive Guerbet test (8) (benzene nucleus)
F ... Negative diazo and Guerbet tests (aliphatic compound)
L ... Starch–iodine test (9) red to yellow (long hydrocarbon chain)
S ... Starch–iodine test blue (short hydrocarbon chain)

A— NO ORGANIC NITROGEN

A I N L	A II N L	A III N L
A I N S	A II N S	A III N S
A I P L	A II P L	A III P L
A I P S	A II P S	A III P S
A I F L	A II F L	A III F L
A I F S	A II F S	A III F S

B— ORGANIC NITROGEN PRESENT

B I N L	B II N L	B III N L
B I N S	B II N S	B III N S
B I P L	B II P L	B III P L
B I P S	B II P S	B III P S
B I F L	B II F L	B III F L
B I F S	B II F S	B III F S

After the main character of the compound has thus been ascertained, the further determination of the nature of the compound is based on the establishment of the group causing solubility in water. Owing to the division into 36 groups, the following separation (Table II) is much simpler than in the old analytical system.

The six AI groups are all sulphonium or phosphonium compounds, so that they may all be classified by the detection of sulphur or phosphorus.

Tests*

Although most of the tests have already been described in the previous paper[1], there have been so many improvements that it is desirable to describe these reactions again. A number of tests have called for further research, the results of which will be given after the description of the tests. In addition several new tests are included. The auxiliary products must first be separated from inorganic salts (see p. 152).

TESTS FOR ORGANIC NITROGEN

(1) *Heating with Alkali*

About 200 mg. of the auxiliary product is introduced into a test-tube with about 300 mg. of sodium hydroxide (in the form of pellets). Into the middle of the test-tube is introduced a small roll of dry surgical gauze, not too tightly rolled. It is advisable to rinse the gauze beforehand with distilled water and dry it, for normal surgical gauze may exhibit an alkaline reaction. On the gauze is placed a piece of red litmus paper moistened with distilled water. During this test the litmus paper is moistened again from time to time with distilled water. The test-tube is supported with the bottom part in a sand-bath at about 160°C. and heated for one hour, the litmus paper being examined to see whether it is turning blue. If so, it is ascertained whether any ammonia is present by means of a drop of Nessler's reagent on a small glass rod.

(2) *Treatment with Alkaline Bromine and Boiling with Alkali*

The auxiliary product (0·8 g.) is dissolved or suspended in 10 c.c. of distilled water, after which 100 c.c. of alkaline bromine is added. This alkaline bromine contains 31·2 g. of bromine and 15·6 g. of sodium hydroxide per litre. After 5 minutes' standing at room temperature, 50 c.c. of sodium hydroxide (300 g./litre) is added, the liquid is boiled for 2 hr. under a reflux condenser, the condenser is removed, and the vapour is tested with red litmus paper. If this paper turns blue, organically bound nitrogen is present. This test is carried out only if the heating with alkali (1) has produced a positive indication of ammonia.

(3) *Boiling with Dilute Alkali*

In some cases it is desirable to know whether a volatile amine has been used as counter-ion in the auxiliary product. In such a case, this may be detected by gently boiling 20 mg. of the auxiliary product with 5 c.c. of 0·05 N. sodium hydroxide in a test-tube for a short time and testing the vapour with red litmus paper.

(4) *Chlorantine Fast Brown BRLL Test*†

The object of this test is to ascertain whether any organic nitrogen is present in the insoluble decomposition product obtained by boiling with sulphuric acid (20). The insoluble decomposition product is boiled (if necessary, after dissolving in ether, decolorising by filtering through active charcoal, and evaporating off the ether) in a test-tube with 10 c.c. of Chlorantine Fast Brown BRLL (200 mg./litre). An intense brown coloration of the upper layer indicates a positive reaction. Care should be taken to ensure that the pH of the boiling liquid is between 4 and 6.

TESTS TO DETERMINE THE IONIC CHARACTER OF THE COMPOUND

(5) *Cation Test*

In the previous paper[1] this test was described as the "Resolin B test". As already hinted at the time and afterwards stated clearly by Wurzschmitt[2], any anion-active substance may be used for this test. However, the reaction is not equally clear with all anion-active substances. The reagents which are suitable for this test are Resoline B (S), Erkantol BX (FBy), Perminal BX (ICI), and Nattal UA (Servo, Delden). To 10 c.c. of the auxiliary product (4 g./litre) is added drop by drop a 10 g./litre solution of one of the above reagents until maximum turbidity or flocculation occurs. Rapid addition of the reagent is to be avoided, since the turbidity may disappear with an excess of reagent. A positive reaction can be confirmed by the bromophenol-chloroform test (28).

(6) *Anion Test*

In the previous paper[1] this reaction was known as the "Solidogen BSE test". This reagent, however, is now practically unobtainable, so that other reagents are required. These must give a very clear solution in water, a requirement which many cation-active compounds fail to satisfy; they must also give a pronounced turbidity with anion-active substances. The reagents satisfying these requirements include the following cation-active compounds— Solidogen FFL (CFM), Fixanol VR (ICI), Liovatin E (S).

The test is carried out in exactly the same way as the cation test. Here, again, it should be noted that the precipitates or turbidity first formed may dissolve in an excess of reagent. A positive result can be confirmed by the Methylene Blue–chloroform test (29).

TESTS TO DETERMINE THE NATURE OF THE HYDROCARBON RESIDUE

Both Wurzschmitt[2] and Bergeron[3] have proposed tests for the hydrocarbon residue different from those used by me. These tests relate in particular to the detection of the naphthalene and benzene nuclei. Before deciding which reactions were to be applied for this purpose, I made a comparative examination with 44 well known alkylnaphthalene-sulphonates, alkylnaphthalenephosphonates, alkylnaphthalene–polyethylene oxide compounds, alkylbenzenesulphonates, and alkylbenzene–polyethylene oxide compounds. This examination related to the nitration and nitric acid tests as carried out by Wurzschmitt, the diazosulphanilic acid test as carried out by Bergeron, and the Guerbet and diazo tests as carried out by van der

† Chlorantine Fast Brown BRLL (Ciba) is a direct dye that is taken up by the weakly cation-active decomposition product (at pH 4–6). Other direct dyes and acid dyes will give in principle the same result, but Chlorantine Fast Brown BRLL is mentioned as being, in this respect, remarkably good.

* More information on Tests 4–7, 9, 16, and 27 is given by van der Hoeve[1], and on Tests 12, 15, 30, and 31 by Wurzschmitt[2].

TABLE II

Identification of Main Groups

(Tests are indicated by numbers in parentheses)

Groups	Test and Result	Inference and Confirmatory Test
AI (all)	(10) Positive	Phosphonium compounds
	(11) Positive	Sulphonium compounds
AIINL, AIINS		Polyethylene oxide–alkylnaphthols (16)
AIIPL, AIIPS		Polyethylene oxide–alkylphenols (16)
AIIFL	(16) Positive—	
	(20) and (21) Positive	Polyethylene oxide esters of fatty acids
	Negative	Polyethylene oxide–alcohols
	Negative	Fatty-acid monoesters of polyhydric alcohols (20) and (21)
AIIFS	(16) Positive	Polyethylene oxide–alcohols
	Negative	Ethers, esters, alcohols, thioethers, and acids
AIIINL, AIIINS	(17) Positive	Alkylnaphthalenephosphonates
	Negative	Alkylnaphthalenesulphonates
AIIIPL, AIIIPS		Alkylarylsulphonates
AIIIFL	(17) Positive—	
	(23) Separated layer or turbid	*sec.*-Alkyl phosphates
	No separated layer or turbidity	*n*-Alkyl phosphates
	Negative—	
	(18) Turbid—	
	(23) Separated layer or turbid	Polymerised ricinoleyl sulphates
	No separated layer or turbidity	
	(22) Turbid	Soaps
	Clear	Low-sulphated fats
	Clear—	
	(26) Alcohols present—	
	(20) Separated layer	Sulphated alkyl ricinoleates
	Turbid or emulsification on washing	"Ricinoleylalkyl ester sulphonates"
	Alcohols absent—	
	(24) Separated layer or turbid—	
	(19) Turbid	Sulphated oleyl acetate
	Clear—	
	(20) and (21) Positive	Highly sulphated fats
	Negative	*sec.*-Alkyl sulphates
	No separated layer or turbidity—	
	(19) Turbid	Alkanesulphonates
	Clear—	
	(20) and (21) Positive	Oleic ester of ethionic acid
	Negative—	
	(16) Positive	Alkylpolyethylene oxide sulphates
	Negative	*n*-Alkyl sulphates
AIIIFS	(26) Alcohols present	Dialkyl sulphosuccinates
	Alcohols absent—	
	(17) Positive—	
	(23) Separated layer or turbid	*sec.*-Alkyl phosphates
	No separated layer or turbidity	*n*-Alkyl phosphates
	Negative—	
	(23) Separated layer or turbid	*sec.*-Alkyl sulphates
	No separated layer or turbidity—	
	(19) Turbid	Alkanesulphonates
	Clear	*n*-Alkyl sulphates
BI (all)*	(12) Positive	Quaternary ammonium compounds
	Negative—	
	(13) Positive	Polyamines
	Negative	Cation-active amines
BIINL, BIINS, BIIPL, BIIPS*	(15) Turbid	Aminopolyethylene oxides (*N* not oxyethylated)
	Clear—	
	(14) Positive	Cation–anion-active compounds
	Negative	Aminopolyethylene oxides (*N* oxyethylated)
BIIFL*	(20) Separated layer	Fatty-acid amide–polyethylene oxides
	No separation—	
	(15) *As Groups BIINL–BIIPS above*	

* Groups BIINL–BIIFL inclusive give a positive reaction in Test (16)

Groups	Test and Result	Inference and Confirmatory Test
BIIFS	(16) Negative	Aminopolycarboxylic acids [e.g. $N(CH_2\text{-}COOH)_3$], amines, amides, etc.
	Positive—	
	(15) Turbid	Aminopolyethylene oxides (N not oxyethylated)
	Clear—	
	(14) Positive	Cation–anion-active compounds
	Negative—	
	(12) Positive	Aminopolyethylene oxides (N oxyethylated)
	Negative	Polyamides (20†) and (21)
BIIINL	(17) Positive	Alkylnaphthalenephosphonates
BIIINS	Negative	Alkylnaphthalenesulphonates
BIIIPL	(16) Positive	Benziminazole derivatives
	Negative	Alkylarylsulphonates
BIIIPS		Alkylarylsulphonates
BIIIFL	(20) No separation—	
	(19) Turbid	Alkanesulphonates
	Clear—	
	(3) Alkaline vapours	*n*-Alkyl sulphates
	No alkaline vapours	Alkylamidoalkanesulphonates
	Separated layer or layer emulsifies on washing—	
	(4) Negative—	
	(25) Positive	Fatty acid–protein condensates
	Negative	Anion-active amines (as AIIIFL)
	(4) Positive—	
	(21) Negative	Anion-active amines having the amino group in the insoluble decomposition product
	Positive—	
	(17) Positive—	
	(23) Separated layer or turbid	Alkylamido-*sec.*-alkyl phosphates
	No separated layer or turbidity	Alkylamido-*n*-alkyl phosphates
	Negative—	
	(21‡) Positive	Alkylamidoalkanecarboxylated salts
	Negative—	
	(23) Separation	Alkylamido-*sec.*-alkyl sulphates
	No separation	Alkylamido-*n*-alkyl sulphates
BIIIFS		Anion-active amines (as AIIIFS)

† Boiling with sulphuric acid for 2 hr.

‡ Test (21) on alcohol extract of the original substance, i.e. not after boiling with sulphuric acid (20)

Hoeve. The nitration test of Wurzschmitt gave 15 wrong results; in particular, with regard to the alkylnaphthalenesulphonates this test was not reliable. The nitric acid test of Wurzschmitt gave a characteristic fluorescence under the ultraviolet lamp in a few cases only. The diazosulphanilic acid test of Bergeron showed a large number of errors. The diazo test of van der Hoeve produced results in agreement with the known composition of the compounds. In two cases the Guerbet test of van der Hoeve gave a negative result where it should have been positive. After the concentration of the sodium nitrite solution had been raised from 1 to 5 g./litre, the results obtained with these compounds also were positive.

Bergeron carried the distinction between the aromatic nuclei even further: he used a ferric chloride test for phenolic compounds and an aluminium chloride–chloroform test for diphenyl compounds. Since I had no auxiliary product with a free phenol group available, the ferric chloride test was always negative. The ricinoleylcresol compounds mentioned in my previous paper[1] were found to be mixtures.

The aluminium chloride–chloroform test was positive with two alkylnaphthalenesulphonates and with the triethanolamine salt of an alkylnaphthalenephosphonate; the other tests were negative.

In view of these results I decided to continue with the diazo and the Guerbet tests, the latter in its improved form.

(7) *Diazo Test*

The neutralised auxiliary product (10 c.c., 4 g./litre) is mixed at room temperature with 10 c.c. of Fast Red Salt AL (NCO) solution (diazotised α-aminoanthraquinone, 20 g./litre). After 15 sec. the colour of the mixture is observed, an orange to brown colour indicating the presence of a naphthalene nucleus.

The azo dye formed is soluble in chloroform. In a few cases, for example when owing to intense turbidity of the auxiliary product it is difficult to judge the reaction, a satisfactory result can be obtained by shaking the reaction liquid with chloroform and observing the colour of the chloroform layer.

(8) *Guerbet Test*

The textile auxiliary product (50–100 mg.) is dried at 110°C. and then heated with 1 c.c. of fuming nitric acid on a sand-bath. The nitric acid is evaporated off, and the residue is taken up in 5 c.c. of 96% ethanol. After this, 1·5 c.c. of

11·5 N. hydrochloric acid is added, and 100 mg. of tin powder introduced into the solution. The mixture is warmed, and after completion of the reduction is cooled to room temperature; water is added to make 20 c.c. and the mixture is filtered. This liquid (5 c.c.) is mixed with 5 c.c. of sodium nitrite solution (5 g./litre) and 5 c.c. of sodium acetate (35 g./litre), and the resulting mixture allowed to stand for 5 min. After this, 5 c.c. of a β-naphthol solution (0·8 g. of β-naphthol + 9·6 g. of sodium hydroxide + 27 g. of sodium acetate per litre) is added. An orange–red coloration after 1–2 min. indicates the presence, a yellow coloration the absence, of aromatic compounds. By the end of the reaction the solution should be alkaline towards Benzopurpurine 4B and acid towards litmus.

(9) *Starch–Iodine Test*

Soluble starch (100 mg.) is dissolved in 10 c.c. of water, and the solution is cooled to room temperature.

The textile auxiliary product (10 c.c., 4 g./litre) is added, and then a sufficient number of drops of 0·01 N. iodine solution for the starch–iodine colour to be produced. A yellow, orange, or red colour indicates a long hydrocarbon chain, a blue colour a short hydrocarbon chain.

TESTS FOR THE SOLUBILISING GROUP

I— CATION-ACTIVE COMPOUNDS

(10) *Test for Phosphorus*

In a small porcelain crucible (about 20 c.c.) there is placed a layer about 3 mm. thick of a finely powdered mixture of 1 part by weight of anhydrous sodium carbonate and 3 parts by weight of sodium nitrate (both reagents should be free from phosphate and sulphate). To this layer is added drop by drop 5 c.c. of a 1% solution of the auxiliary product in methanol, and then the same quantity of the soda–nitrate mixture as before. The crucible is placed on a pipeclay triangle, and the methanol vapours are ignited with a strip of paper impregnated with alcohol. After combustion of the methanol, the mixture is heated over a gas flame (which should not extend above the rim of the crucible) until a clear melt has been obtained. The melt is then allowed to cool, 10 c.c. of distilled H_2O added, and the solution boiled over a small flame and filtered. The filtrate is subjected to the sulphomolybdate test (17).

(11) *Test for Sulphur*

The filtrate from the dissolved soda–nitrate melt of the auxiliary product (10) is acidified with nitric acid (100 g./litre), and tested for sulphate with barium chloride.

(12) *Permanganate–Chloroform Test*

To 4 c.c. of a 1% aqueous solution of the auxiliary product is slowly added 0·05 N. potassium permanganate solution. This may give rise to a brown–red or red–violet flocculation or turbidity. The potassium permanganate solution is added until this flocculation or turbidity is at its maximum. The mixture is then shaken for a short time with 3 c.c. of chloroform. If the chloroform layer is coloured red–violet or red–brown, this test for quaternary ammonium, quaternary phosphonium, or ternary sulphonium compounds is positive.

(13) *Sodium Nitroprusside Test*

To 10 c.c. of a 0·4% solution of the auxiliary product is added drop by drop a freshly prepared 5% solution of sodium nitroprusside.

An intense orange turbidity or an orange precipitate indicates the presence of phosphonium, sulphonium, or quaternary ammonium compounds or polyamines.

II— NON-IONIC COMPOUNDS

(14) *Acid Cation Test*

To 5 c.c. of a 0·4% solution of the auxiliary product is added 5 c.c. of 0·1 N. hydrochloric acid, and then the cation test (5) is carried out.

Owing to the lowering of the pH of the solution the cation-active properties of the cation–anion-active compounds become more pronounced.

(15) *Heating with Salt*

A 0·4% solution of the auxiliary product (10 c.c.) is mixed with 3–5 c.c. of a 10% solution of ammonium chloride. If the solution remains clear at room temperature, it is heated. A turbidity at room temperature or elevated temperature indicates the presence of polyethylene oxide derivatives which are not oxyethylated at the quaternary nitrogen atom. This inference can be drawn here because the preceding tests have already shown that the compounds concerned are non-ionic products. If a mixture of auxiliary products is being investigated, one has to be cautious in making this inference, since several cation- and anion-active compounds are not sufficiently soluble in the salt solution.

(16) *Potassium Iodide–Iodine Test*

The auxiliary product (5 c.c., 4 g./litre) is mixed at room temperature with 10 c.c. of iodine solution (1·27 g. of iodine plus 2·0 g. of potassium iodide per litre). A red–brown or grey coloration indicates a polyethylene oxide derivative or a compound having a cation-active group in the molecule.

III— ANION-ACTIVE COMPOUNDS

(17) *Sulphomolybdate Test*

The auxiliary product (10 c.c., 4 g./litre) is neutralised with 0·1 N. sulphuric acid until the red colour of phenolphthalein just disappears. Sodium chloride (300 mg.) is added, and then three drops of sulphomolybdate solution (10 g. of ammonium molybdate plus 300 c.c. of sulphuric acid (50% by vol.) per litre) and a strip of tin foil. The mixture is shaken for a moment, and after 30 min. it is examined to see whether a blue colour appears.

It should be noted that the presence of polyethylene oxide derivatives may cause an apparently positive reaction for phosphate. If mixtures containing polyethylene oxide derivatives are being investigated, it is preferable first to identify phosphorus according to Test (10).

(18) *Sulphuric Acid Test*

The auxiliary product (10 c.c., 4 g./litre) is mixed at room temperature with 10 c.c. of sulphuric acid (10 g./litre). After standing for 2 hr., the result is assessed.

(19) *Magnesium Sulphate Test*

The textile auxiliary product (10 c.c., 4 g./litre) is mixed at room temperature with 10 c.c. of magnesium sulphate solution (20 g. of $MgSO_4$, $7\,H_2O$ per litre). After 2 hours' standing the effect is observed. If the auxiliary product does not form a perfectly clear solution in water, a comparison with the 1 : 1 solution of auxiliary product diluted with distilled water will give an indication.

(20) *Boiling with Sulphuric Acid*

The textile auxiliary product (1 g.) is boiled vigorously for 30 min. with 40 c.c. of sulphuric acid (100 g./litre), under a reflux condenser. Any product that may be precipitated is separated from the sulphuric acid layer after cooling, by means of a separating funnel or a filter, and washed at least three times with distilled water. (The pH of the last lot of rinsing water should be >4.)

(21) *Fall in pH*

The upper layer from the treatment with boiling sulphuric acid (20), which should weigh at least 150 mg., is shaken with about 20 c.c. of ethanol (50% by vol.), and to this are added 2 drops of methyl red solution. The mixture is titrated, with continuous shaking, with 0·1 N. sodium hydroxide until the colour of the solution is yellow; after this, 2 drops of phenolphthalein solution are added, and it is ascertained how much 0·1 N. sodium hydroxide is required to obtain a red coloration. If more than 1·5 c.c. of sodium hydroxide has to be added, the result is considered positive.

(22) *Caustic Soda Test*

The auxiliary product (10 c.c., 4 g./litre) is mixed with 10 c.c. of sodium hydroxide solution (50 g./litre). Immediately after mixing at room temperature the result is assessed. A precipitate or intense turbidity indicates soap.

(23) *Heating Test*

The textile auxiliary agent is heated, in the undiluted state, for 24 hr. in an open test-tube at 110°C. It is ascertained whether the originally readily soluble product is still soluble in boiling water.

Secondary alkyl sulphates and secondary alkyl phosphates are not stable under these conditions. During the decomposition the pH is very greatly lowered. In cases where the result of the test is not quite certain, checking the pH may give a definite indication.

(24) *Acid Heating Test*

This test is carried out in exactly the same way as the preceding one (23), but the pH of the solution of auxiliary product is first adjusted to 5·0. This is done to prevent any –COONa groups present in the product from acting as a pH buffer. In fact, the decomposition of the auxiliary product proceeds autocatalytically under the influence of the hydrogen ions formed.

(25) *Biuret Test*

The textile auxiliary product (10 c.c., 4 g./litre) is mixed with 10 c.c. of sodium hydroxide (10 g. per litre) and 1·5 c.c. of copper sulphate solution (3 g. of $CuSO_4,5H_2O$ per litre). After gentle heating, a violet coloration indicates a positive result.

SPECIAL TESTS

(26) *Alkaline Distillation*

The original textile auxiliary product (5 g.— not the alcoholic extract) is boiled, after steaming off any solvents present, for 2 hr. with 60 c.c. of sodium hydroxide (300 g./litre) under a reflux condenser. The liquid is then distilled, and the distillate examined for alcohols. This is done by oxidising the distillate with chromic acid (50 g. $K_2Cr_2O_7$ + 65 g. H_2SO_4 per litre) and redistilling. This last distillate is mixed with carbazole–sulphuric acid (2 g. of carbazole to 1 litre of nitrate-free concentrated sulphuric acid). A few drops of the distillate to 5 c.c. of carbazole–sulphuric acid are sufficient for the test. Here it is also possible to get some idea of the nature of the alcohols present in the auxiliary product. In this test methanol gives a blue coloration; ethanol a wine-red; *iso*propanol, *iso*butanol, and pentanol a red–brown; and 2-ethyl-1-hexanol a greyish-yellow coloration.

(27) *Ammonium Cobaltithiocyanate Test*

For the analysis of the single compounds the importance of this test has greatly decreased. In the analysis of mixtures, however, it can be quite useful for the detection of mixtures of cation-active substances with polyethylene oxide derivatives.

The textile auxiliary product (5 c.c., 4 g./litre) is mixed at room temperature with 5 c.c. of ammonium cobaltithiocyanate solution (174 g. of ammonium thiocyanate and 2·8 g. of cobalt nitrate per litre). The mixture is shaken vigorously, and after 2 hr. the colour of the liquid (not of the precipitate) is observed. If the colour is red–violet to violet, the reaction for polyethylene oxide compounds is negative; if the colour is blue, the reaction is positive. A blue precipitate indicates the presence of cation-active compounds.

(28) *Bromophenol Blue–Chloroform Test*

The auxiliary product (10 c.c., 4 g./litre) is mixed with 0·1 c.c. of an ethanol solution of bromophenol blue (1 g./litre) and 0·1 c.c. of soda (100 g./litre). After this, 0·5 c.c. of chloroform is added, and the mixture is shaken vigorously. A blue–violet coloration of the chloroform indicates a positive reaction.

(29) *Methylene Blue–Chloroform Test*

The auxiliary product (5 c.c., 4 g./litre) is shaken with 5 c.c. of methylene blue solution (0·1 g./litre) and 1 c.c. of chloroform. A blue coloration of the chloroform layer indicates a positive reaction.

(30) *Heliogen Blue SBL Test*

To 10 c.c. of auxiliary product (4 g./litre) Heliogen Blue SBL* solution (2 g./litre) is gradually added, the precipitate formed is filtered off, and the filtrate is subjected to the anion test (6). It is necessary to ensure that no excess of the blue is used, and this is tested by spotting on filter paper, when a blue rim should just not appear.

(31) *Blancotan B Test*

To 10 c.c. of the auxiliary product (4 g./litre) is added a sufficient quantity of Blancotan B (Basf)† (50 g./litre) to prevent any further turbidity or precipitation. After this, 10 c.c. of 0·01 N. iodine in potassium iodide is added. A dark brown precipitate or dark brown suspension indicates polyethylene oxide derivatives.

Examination of Mixtures

In commerce there occur mixtures of auxiliary products, the object of the mixing being to reinforce in some way or other the action of the main component or to reduce or eliminate any disagreeable side-effect. In principle the main component may be mixed with one of the following subsidiary components— (i) inorganic salts, (ii) organic solvents, (iii) some other typical textile auxiliary product.

(i) MAIN COMPONENT AND INORGANIC SALTS

The principle of the separation is drying, followed by extraction with methanol or ethanol and drying again.

The drying may, however, give rise to complications, since secondary alkyl sulphates and phosphates decompose on being dried at elevated temperatures (e.g. 100°C. and above). If such phenomena occur, the drying will have to be effected at 50°C.

With many polyethylene oxide compounds ether may be used instead of methanol or ethanol.

(ii) MAIN COMPONENT AND ORGANIC SOLVENTS

Before the tests for the main component are carried out, the latter should be freed from the solvent by steam distillation. Solvents may affect various reactions by making it impossible to judge the turbidity. They may also affect the starch–iodine test (9). Alcohols with a moderately long hydrocarbon chain, e.g. 2-ethyl-1-hexanol, in spite of their low solubility in water, shift the blue starch–iodine colour toward violet. In the alkaline heating test (1) they may cause creeping of the caustic soda, and thus suggest a positive reaction. Any excess of solvents, therefore, must be removed before this test.

(iii) MAIN COMPONENT AND A SECOND TEXTILE AUXILIARY PRODUCT

In this case there occur numerous complications, most of which cannot be predicted. Many reactions for the main component may be interfered with by the admixture. Further, if a mixture exhibits a positive reaction, one cannot be sure whether this reaction is caused by the main or by the secondary component.

* Heliogen Blue 3BL is an anionic dye.

† An aluminium compound, known also as "Blancorol B".

With a two-component system the main classification at once becomes much more complex. In order not to make this too complicated, polyethylene oxide derivatives alone have been taken into account for the non-ionic component, a simplification which fits in well with present-day practice.

If the mixture does not contain any organic nitrogen, the system is A + A (cf. Table I).

If the mixture does contain organic nitrogen, the system may be either A + B, B + A, or B + B.

The detection of the organic nitrogen is not affected by admixture of a second textile auxiliary product.

The possible types of binary mixtures of textile auxiliary products are set out in Table III, the main component being given first and the secondary component in parentheses.

TABLE III

Classification of Binary Mixtures of Auxiliary Products

Cation Reaction (5) positive	Cation Reaction (5) negative Anion Reaction (6) negative	Anion Reaction (6) positive
I + (I)	II + (I)	III + (I)
I + (II)	II + (II)	III + (II)
I + (III)	II + (III)	III + (III)

The presence of polyethylene oxide derivatives in mixtures affects both the cation and the anion reactions, the extent of this influence depending largely on the nature of the polyethylene oxide compound. Even an addition of 30% of polyethylene oxide derivative may cause the test results to become negative, and an addition of 50% of polyethylene oxide derivative in most cases suppresses the cation or the anion reaction.

Fortunately, however, there are two other tests which are only slightly affected by polyethylene oxide derivatives. These are the bromophenol blue–chloroform test (28)[7] and the methylene blue–chloroform test (29)[8]. An admixture of 5% of cation-active substance to a polyethylene oxide compound can be detected by means of the bromophenol blue–chloroform test, and so can $< 2\%$ of anion-active substance by means of the methylene blue–chloroform test.

With the aid of these tests the groups II + (I), II + (II), and II + (III) can therefore be separated.

A second separation is possible with the groups I + (I), I + (II), and I + (III). In this case we can make use of the ammonium cobaltithiocyanate test (27). This test gives a blue precipitate and a violet solution with all cation-active auxiliary products; with the polyethylene oxide derivatives a blue solution is obtained. It has now been found that this test gives a blue precipitate and a blue solution with mixtures of 93% of cation-active substance and 7% of a lauryl polyethylene oxide containing four ethyleneoxy groups. For mixtures of cation-active substances with anion-active substances the Heliogen Blue SBL test (30) of Wurzschmitt[9] gives some indication. If the mixture contains two components, with polyethylene oxide

derivatives admixed, the Heliogen Blue SBL test does not give any indication, because no filterable precipitate of the cation-active substance with the Heliogen Blue is then formed. Even 20% of active polyethylene oxide derivatives may greatly interfere with the reaction.

The groups III + (I), III + (II), and III + (III) also present many difficulties. With the aid of the Heliogen Blue SBL test the group III + (I) can be distinguished. It is much more difficult to detect polyethylene oxide derivatives in admixture with an anion-active substance. The potassium iodide–iodine test (16), for example, is greatly affected by an excess of anion-active substance, especially if the latter has a long hydrocarbon chain and the ethylene oxide chain in the polyethylene oxide compound is short. In spite of this, this test can generally be used to identify polyethylene oxide compounds in anion-active auxiliary products. If conditions are favourable, 4% can be detected; if they are unfavourable, it is not possible to detect less than 14%. Destruction of the anion-active substance by boiling with sulphuric acid makes the test much more sensitive.

The ammonium cobaltithiocyanate test (27) is much more sensitive still to the presence of anion-active auxiliary products. In an anion-active auxiliary product having a short hydrocarbon chain 10% of polyethylene oxide compound can just be detected with the aid of this test. In an anion-active auxiliary product with a long hydrocarbon chain this limit is 14–50%!

This difference in behaviour between tests (16) and (27) makes it possible in some cases to distinguish a mixture of an alkyl sulphate with a polyethylene oxide compound from an alkyl-polyethylene oxide sulphate, since with the latter compound the ammonium cobaltithiocyanate test (27) is not affected as greatly as the potassium iodide–iodine test (16).

Another method for the detection of polyethylene oxide derivatives in anion-active mixtures consists in precipitating the major part of the anion-active substance with Blancotan B[10] and testing with potassium iodide–iodine (31). A dark brown precipitate or a dark brown suspension indicates the presence of a polyethylene oxide derivative.

Nature of the Hydrocarbon Chain

The diazo test (7) is positive if only 10% of alkylnaphthalene-sulphonate or -phosphonate or alkylnaphthol–polyethylene oxide is present.

The Guerbet test (8) gives a positive result even with a low percentage of alkylaryl compounds. The starch–iodine test (9), with mixtures of compounds having long and short hydrocarbon chains respectively, gives a result which is mainly determined by the compound with the long hydrocarbon chain. A large proportion of a compound having a short hydrocarbon chain, mixed with a small proportion of a compound having a long hydrocarbon chain, may produce an intermediate colour (e.g. violet).

On the basis of the above considerations, a scheme of analysis of mixtures (Table IV) has been worked out.

TABLE IV

Analysis of Mixtures

System	Test and Result	Type of Mixture
A + A		
Cation-active	No further separation necessary, since sulphonium and phosphonium compounds so rare in commerce that mixtures unlikely	
Non-ionic	(29) Positive	II + (III)
	Negative	II + (II)
Anion-active	(31) Positive	III + (II)
	Negative	III + (III)
B + A, A + B, or B + B		
Cation-active	(27) Blue ppte. and blue soln....	I + (II)
	Blue ppte. and red–violet soln.—	
	(30) Positive	I + (III)
	Negative	I + (I)
Non-ionic	(28) Positive	II + (I)
	Negative—	
	(29) Positive	II + (III)
	Negative	II + (II)
Anion-active	(30) Positive	III + (I)
	Negative—	
	(31) Positive	III + (II)
	Negative	III + (III)

This main separation is followed by the tests for the hydrocarbon chain, which in any case determine the character of one of the compounds.

In this way we obtain a good deal of information about the character of the mixture. In order to get some more detailed information about the mixture the following tests may be useful—

In the presence of an excess of quaternary ammonium bases cation-active mixtures exhibit a positive permanganate–chloroform reaction (12).

If polyethylene oxide derivatives are present, non-ionic mixtures give a positive iodine–potassium iodide reaction (16).

Anion-active mixtures may give rise to very great difficulties during further examination. In fact, many reactions may be seriously affected.

Some of the tests, and the interfering effects that may occur in these cases, may be described as follows—

1. DETECTION OF PHOSPHORUS (10)—It is dangerous to apply the sulphomolybdate test directly, for polyethylene oxide derivatives exhibit a positive sulphomolybdate reaction.

2. DETECTION OF SULPHUR (11)—This reaction is not affected.

3. HEATING TEST (23)—Where mixtures are concerned, it is quite possible for the decomposition product of *sec.*-alkyl sulphates or phosphates to be emulsified by the stable admixture. In such a case it is of the greatest importance to investigate the lowering of the pH.

4. ACID HEATING TEST (24)—Just as with test (23) above, the lowering of the pH is again the most important phenomenon.

5. BOILING WITH SULPHURIC ACID (20)—The results can be classified into three groups—(*a*) the reaction liquid remains clear; (*b*) turbidity or emulsification occurs; (*c*) precipitates appear. In the first case both components of the mixture are stable towards acid boiling; in the second case one

of the two components is stable; in the third case either both components are unstable or an excess of an unstable component is present.

6. Fall in pH (21)— If upon boiling with sulphuric acid a clear liquid is obtained, the determination of the change in pH is unnecessary. If intense turbidity or emulsions appear, the whole of the liquid after boiling should be made neutral to methyl red, and the quantity of alkali required to turn phenolphthalein red should be determined. A precipitate which does not emulsify on washing should be treated in the same way as the single compounds.

7. Chlorantine Fast Brown BRLL Test (4)— This test can be applied only if a precipitate is formed on boiling with sulphuric acid. Owing to the tendency to add more stable to less stable compounds in mixtures, this case occurs much less frequently than with the single compounds.

8. Boiling with Dilute Alkali (3)— If a volatile amine is used as counter-ion, a positive result will be obtained also with mixtures.

9. Alkaline Distillation (26)—This test, too, gives a positive result with mixtures, if ester sulphates or ester sulphonates of volatile alcohols are used.

With the aid of these tests a tolerably good preliminary idea is gained of the composition of the anion-active mixture. It has thus been ascertained whether or not the mixture contains primary or secondary alkyl phosphates, secondary alkyl sulphates, carboxylate salts, ester sulphates, or ester sulphonates. In a few cases the presence of fatty-acid amido-sulphates or -phosphates can be detected. Further, there are some indications as to the presence of polyethylene oxide derivatives, cation-active compounds, naphthalene or benzene nuclei, and the presence or absence of hydrocarbons with a long hydrocarbon chain. It is known whether a volatile amine has been used as counter-ion.

From a combination of these data a good deal can be inferred about the nature of the mixture.

If an unknown substance is examined according to the scheme for unmixed auxiliary products and the textile properties of this substance are found to differ from those which might be expected from the result, it is desirable to consider the result in the light of the mixture examination. I am quite aware that I cannot give a complete guide for mixtures. The above will, however, be a valuable aid, while it will be necessary to carry out research on one's own account so as to try to gain further information in any particular case.

Nico ter Kuile en Zonen NV.
Enschede
Holland

(*Received 11th March 1953*)

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[10] Idem, *ibid.*, **130**, 117 (1950).

Notes

Meetings of Council and Committees

March

Perkin Centenary— 8th
Council— 10th
Publications— 16th
Colour Index Editorial Panel— 25th
Fastness Tests Co-ordinating— 30th

Proceedings of the Council

At meetings of the Council held at the offices of the Society, 19 Piccadilly, Bradford 1, on 13th January, 17th February, and 10th March 1954, the proceedings included the following items of interest—

B.S.I. Committee on Discharge Lamps— Mr. J. C. Guthrie was nominated to represent the Society on Technical Committee LGE/6/6— Discharge Lamps— of the British Standards Institution.

A.A.T.C.C. Perkin Centennial Committee— The recommendation of the Perkin Centenary Committee, that the Society be represented on the above committee by the President, was endorsed.

Mercer Lecture Discussion— The unanimous recommendation of the Mercer Lecture Committee, that there should be no discussion after the lecture, was accepted.

London Lecture— The President reported that the second London Lecture, held on 5th March 1954, had been a great success. Mr. Donald King, of the Victoria and Albert Museum, had given a very interesting and fully illustrated survey of *The History and Development of Textile Printing in London and the Home Counties.*

Membership— Fifty-seven applications for ordinary membership and ten for junior membership were approved. Messrs A. Crosby, V. Chipalkatti, and J. Z. Sadowski were readmitted to membership.

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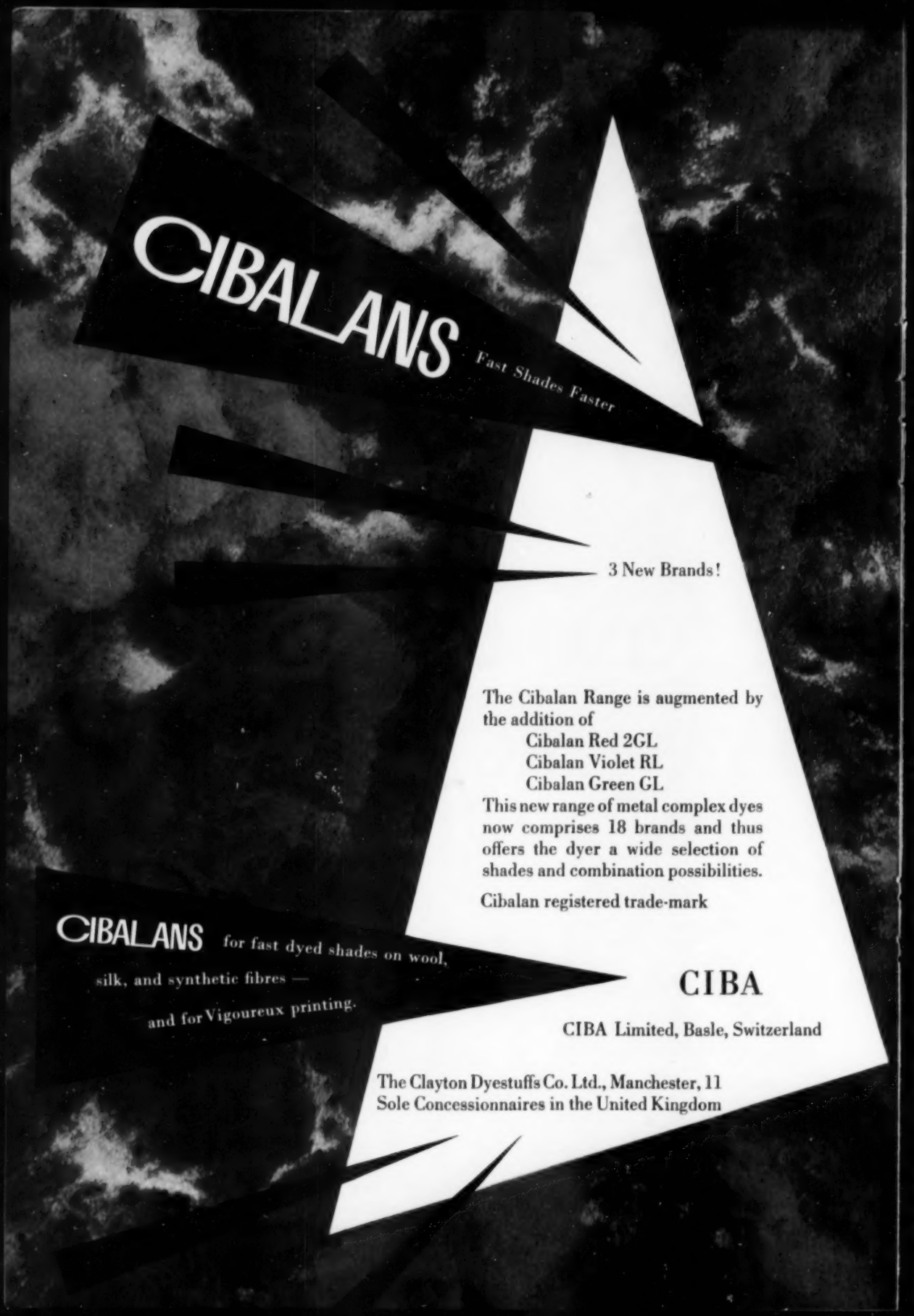
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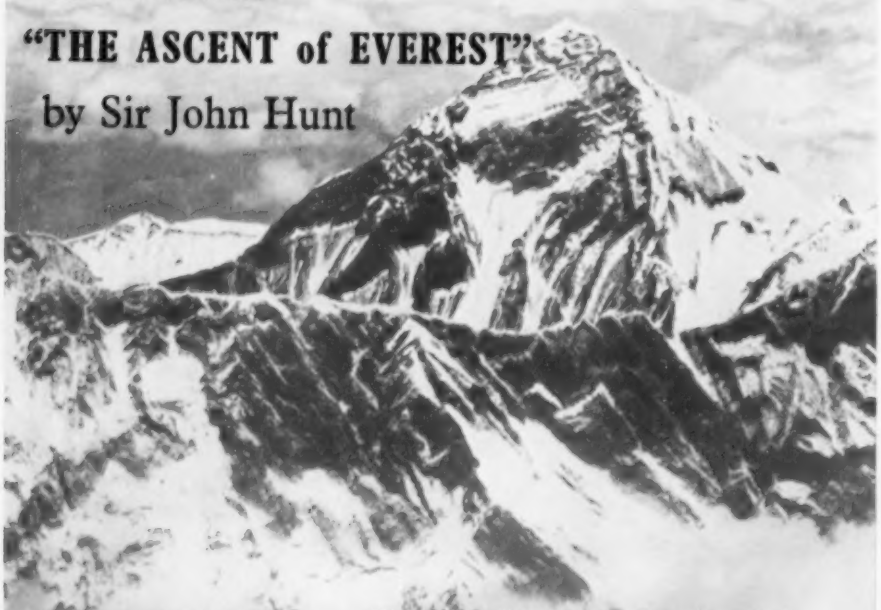

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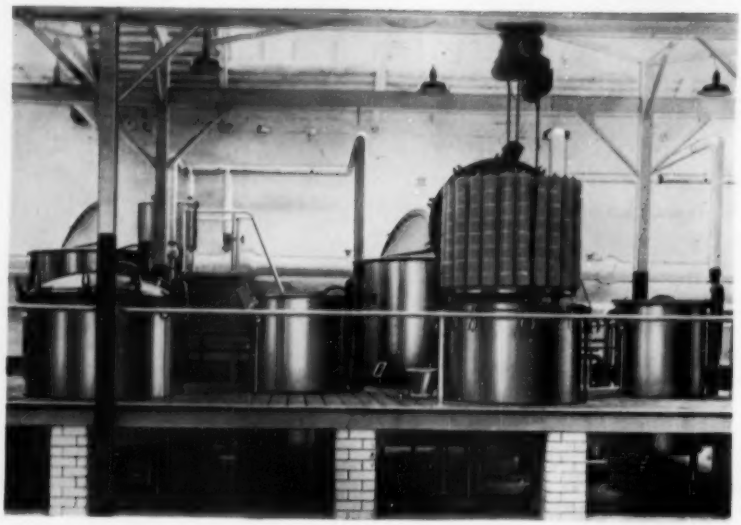

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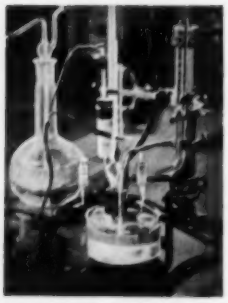

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Award of Fellowships

With the inception of the Diplomas Scheme it became necessary for Council to elect from the membership a nucleus of Fellows so that candidates for the Associateship should have ready approach to the three Fellows necessary to support their application. It was recognised that the inclusion of a member's name in the first list of Fellows would be a mark of signal distinction, and after careful and prolonged consideration it was decided that the list should be confined to those who had either—

(*a*) Received from the Society, or been nominated by the Society to receive, a reward for work which had advanced the knowledge of tinctorial technology or had been a co-author of such work; or

(*b*) Served on one of the Society's technical committees, e.g. the Fastness Tests Co-ordinating Committee; or

(*c*) Otherwise been invited by the Society to undertake work for which high professional qualifications in tinctorial technology were necessary,

it being felt that those included in these categories had already received the Society's recognition of their professional standing.

The following have therefore been elected Fellows of the Society—

G. B. Angus	F. F. Elsworth	J. T. Kidd	J. Starkie
W. Armfield	W. W. England	W. Kilby	E. Stead
C. P. Atkinson	A. E. Everest	B. Kramrisch	C. B. Stevens
T. Barr	F. Farrington	G. Landells	J. W. Strudwick
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H. Blackshaw	R. K. Fourness	L. L. Lloyd	J. V. Summersgill
J. Boulton	M. R. Fox	N. G. McCulloch	H. A. Thomas
H. H. Bowen	J. H. Garner	R. A. McFarlane	A. Thomson
W. Bradley	W. Garner	K. McLaren	D. Traill
G. G. Bradshaw	R. E. Garrod	J. T. Marsh	E. R. Trotman
H. à Brassard	C. H. Giles	R. H. Martin	H. A. Turner
A. Breare	A. Goddard	A. B. Meggy	A. G. Tyler
P. W. Carlene	F. L. Goodall	T. H. Morton	T. Vickerstaff
D. Carter	J. M. Goodall	E. F. Nattrass	J. A. Wallwork
E. G. H. Carter	J. G. Grundy	H. C. Olpin	E. Waters
C. O. Clark	N. Hamer	W. Parry	C. S. Whewell
A. Clayton	H. Hampson	R. Pendlebury	G. S. J. White
E. Clayton	R. J. Hannay	W. Penn	A. Whitehead
A. J. Crockatt	F. C. Harwood	R. H. Peters	C. M. Whittaker
C. J. T. Cronshaw	J. M. Hay	H. Phillips	C. C. Wilcock
H. Cunliffe	T. F. Heyes	J. A. Potter	G. M. Williams
P. W. Cunliffe	G. S. Hibbert	J. M. Preston	E. Wilson
D. A. Derrett-Smith	H. H. Hodgson	E. Race	E. R. Wiltshire
G. T. Douglas	J. Holt	J. W. Reidy	S. G. A. Wise
A. W. Doyle	G. G. Hopkinson	E. Rolfe	F. C. Wood
W. A. Edwards	E. Isles	F. Scholefield	G. F. Wood
R. L. Elliott	H. Jennison	F. Smith	H. Yeoman
H. W. Ellis	W. Kershaw	J. B. Speakman	J. A. Young

It will be appreciated that the principle employed in forming this list made it almost inevitable that it would be confined to members normally resident in the United Kingdom. Recognition of overseas members is receiving earnest consideration and a list of overseas Fellows will be published in due course.

Deaths

We regret to report the loss by death of Messrs. R. P. Foulds, T. Gregson, F. Oxley, W. Oxley, H. B. Rowe, and C. R. Thitchener.

Perkin Medal

On the recommendation of the Society's Medals Committee, Council has decided to award to Dr. Arthur Zitscher the Perkin Medal for his work leading to the discovery of the arylamides of 2 : 3-hydroxynaphthoic acid.

Dyers and Colourists Association of South Africa

Council has been very happy to accede to the request for affiliation of the newly formed Dyers and Colourists Association of South Africa, and takes this opportunity of wishing the Association a very successful future.

The Chairman of the Association is Joseph H. Allen, Esq., 1007–8 Salisbury House, West Street, Durban.

Association of British Chemical Manufacturers

The new address of the A.B.C.M. and affiliated associations operated by A.B.C.M. staff is Cecil Chambers, 86 Strand, London W.C.2. The telephone number is COVent Garden 2363 (4 lines), and the telegraphic address Manuchem, Rand, London.

West German Production of Dyes in 1953

Production of dyes in Western Germany in 1953 was approx. 37,000 tons, being 54% more than in 1952 and valued at 480 million DM.

C O C

World Congress on Surface-active Agents

Paris, 30th August to 3rd September 1954

It is proposed that different sections of the congress will be devoted to physical chemistry, analysis, test methods, production, and applications, including dyeing, printing, laundering, and scouring. The venue will be the Sorbonne. Further information can be obtained from the Secrétariat général du Ier Congrés mondial de la Détergence, 70 Champs-Elysées, Paris VIII, France.

International Congress of Man-made Textiles

Paris, 31st May to 3rd June 1954

The theme of this congress will be *Textiles of Tomorrow*, its aim being to bring together the producers and the users of man-made textiles. The congress will be held at the Centre "Marcelin Berthelot", 28 rue Saint-Dominique, Paris. Full details can be obtained from the Comité International de la Rayonne et des Fibres Synthétiques, 18 rue d'Aguesseau, Paris VIII, France, or from the British Rayon and Synthetic Fibres Federation, Hamilton House, 138 Piccadilly, London W.1.

Japanese Production of Dyes in 1953

According to the Japanese Ministry of International Trade and Industry, Japanese production of dyes in 1953 was 19,400 tons compared with 13,700 in 1952 and 14,100 in 1951. C O C

India's Capacity for Synthetic Dye Production

According to the Indian Tariff Commission, India's present annual capacity for the manufacture of synthetic dyes is in million lb. : azo dyes 2·2; sulphur black 1; azoic compositions— rapid fast types 0·4, Rapidogen type 0·152; azoic diazo components— fast colour salt type 0·997; solubilised vat dyes 0·15; and others 0·079. The Indian synthetic dye industry is, and as far as can be foreseen is likely to remain, dependent almost entirely upon imported intermediates. C O C

British Production of Rayon and Synthetic Fibres in 1953

Total production of rayon and synthetic fibres in the U.K. established a new record in 1953, being at 419,000,000 lb., 9% more than the previous record set up in 1951 and 48% more than the 1952 production. Staple fibres accounted for 202,000,000 lb. of the production and continuous filament for 217,000,000 lb. C O C

Papers read at the Huddersfield Dyers' Mutual Improvement Society

Mr. F. Wilkinson has given to the Society a copy of the very scarce book *An Abridged Report of Papers Read at the Huddersfield Dyers' Mutual Improvement Society* Huddersfield, 1872. It is a book of 37 pp. and the notice on the verso of the title page is well worth quoting in full as this book is a direct ancestor of this *Journal* and the spirit which possessed those responsible for the publication of this little book still animates our Society. "The following pages result from the endeavours of a few illiterate individuals who, feeling their need of improvement, have by the means of discussions, and the reading of papers during the winter months endeavoured to improve themselves and believing that many things that had been brought forward at their meetings which might otherwise be forgotten, it was determined to have an abstract of the papers published, for the benefit of the members and other friends who wished to possess them". The contents of the book comprise: *Introductory Paper* by J. B. Wilkinson, who also contributed two other papers, viz. *Catechu : Its Preparation and Properties* and *On Fustic*; four papers by Joe Frost, viz. *Suitability of Water for Dyeing and Printing, Scouring, Chromium and its Compounds* and *On Mordants*; *Blue Vitriol* by Dan Schofeld, *Indigo* by David Spivey, and *Camwood* by C. Challenger. C O C

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Ultrasonics in Industry—A Critical Assessment. II—Applications. M. H. M. Arnold. *Chem. and Process Eng.*, **35**, 15–19 (Jan. 1954).

The industrial application of ultrasonic vibrations is discussed briefly under headings which include the following—flaw detection and thickness measurement, viscometry, emulsification, solid wetting and dispersion, laundering, de-dusting of gases, coagulation in liquids, flocculation, scale prevention, and a number of physico-chemical effects such as the disruption of micellar structure in soln. of high polymers, and the degradation of proteins, polysaccharides, and synthetic polymers. The scope and limitations of the methods are clearly stated. There is a useful bibliography. J. W. D.

From Ethyl Alcohol to Cellulose Acetate—Australia's New Chemical Industry. *Chem. and Process Eng.*, **35**, 5–9, 19 (Jan. 1954).

A detailed account of the new factory erected by C. S. R. Chemicals Ltd. at Rhodes, New South Wales, for the production of chip-form cellulose acetate. The necessary plant is embodied for the conversion of ethanol (produced by the fermentation and distillation of molasses) to acetic acid and acetic anhydride; a number of other organic chemicals are also produced from the molasses. Each stage of the manufacture is fully described, and there are several photographs and flow-sheets. J. W. D.

PATENT

Nylon Preboarding Machine. Dexdale Hosiery Mills. *BP* 702,626

The machine embodies the main features described in *BP* 568,946 but in addition incorporates means for circulating hot air within the chamber thus enabling drying to follow the steam setting of the hose. Alternatively the machine may be operated entirely for either successive steaming or drying. Air induced into the chamber and circulated during the drying period is heated by closed steam coils mounted within the chamber. G. E. K.

II—WATER AND EFFLUENTS

Water Treatment—Tannery Waste. B. A. Southgate. *J. Soc. Leather Trades Chem.*, **37**, 393–399 (Dec. 1953).

Tannery waste liquors present a serious problem with regard to effluent purification, partly because they contain both sulphides and organic matter, and satisfactory treatments are still lacking. Some methods used in other difficult circumstances are surveyed, and it is suggested that a more satisfactory approach would be to reconsider the processes involved in the leather treatment, thus perhaps reducing the troublesome nature of the raw effluent, rather than to devise purification methods entirely on the basis of existing effluents. J. W. B.

Waste Treatment and Stream Pollution—a Glossary of Terms. A.A.T.C.C. Rhode Island Sectional Stream—Pollution Committee Report. *Amer. Dyestuff Rep.*, **42**, P695–P706 (12 Oct. 1953).

In-plant Process Control for the Reduction of Waste. R. H. Souther. *Amer. Dyestuff Rep.*, **42**, P656–P664 (28 Sept. 1953).

A brief survey of effluent treatment in the factory emphasises the advantages of using less chemicals in textile processing, and of increasing the economy in effluent treatment by predetermination of the quantity of purifying agents required. J. W. B.

Ion Exchange and its Applications in the Fine Chemical Industry (XIV p. 172).

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Wool Wax. V—The Constitution of the α-Hydroxy Acids derived therefrom. D. H. S. Horn, F. W. Hougen, E. von Rudloff, and D. A. Sutton. *J.C.S.*, 177–180 (Jan. 1954).

The saponifiable fraction of wool wax from merino wool is found to yield nearly 30% of a mixture of optically active 2-hydroxy-*n*-dodecanoic, -*n*-tetradecanoic, -*n*-hexadecanoic, -*n*-octadecanoic, and -16-methylheptadecanoic acid. H. H. H.

Improvement of the Dirt-suspending Power of Carboxymethyl Cellulose. K. J. Nieuwenhuis. *J. Polymer Science*, **12**, 237–252 (Jan. 1954).

The salts of carboxymethyl cellulose (CMC) owe their dirt-suspending power to the fact that they are absorbed from aqueous solution by cellulose. To exert their greatest effect their degree of substitution should be about 0·5, the substitution should be as uniform as possible and the cellulose chains should be neither too short nor too long. The fibre contents, i.e. the percentage of insoluble and partly swollen fibres in 0·05N-NaOH can be taken as a measure of the uniformity of substitution. Technical products differ greatly in pH and salt contents: therefore, the viscosity of their solutions containing 1% of the cellulose ether, 2·5% Na_2CO_3 and 2·5% $NaHCO_3$, is taken as a measure of the length of their cellulose chains. The easiest way to obtain uniformly substituted products is to disperse cellulose and reagents in an alcohol or hydrocarbon, fibre contents of $< 4\%$ being obtained. With such a product dirt-suspending power was greatest with a degree of substitution of 0·45–0·53 and a viscosity, measured as above with a Hoeppler standard viscometer, of 11–29 c.p.s. Substitution in general is much more uneven when the CMC is prepared according to the customary method by mixing cellulose, NaOH and $ClCH_2COONa$ or $ClCH_2COOH$ and a little water. With such a product dirt-suspension was best with an average degree of substitution of 0·51–0·60, fibre contents 6–16% and viscosity 20–30 c.p.s. C. O. C.

Constitution of Sulphated Oils. V—Effects of Variations in Preparation on the Composition of Sulphated Oleic Acid. D. Burton and L. F. Byrne. *J. Soc. Leather Trades Chem.*, **37**, 321–330 (Oct. 1953).

Recent Developments in the Study of Oxidative Deterioration of Lipids. C. H. Lea. *Chem. and Ind.*, 1303–1309 (5 Dec. 1953).

A survey in which is advanced a possible derivation from the three isomeric linoleate hydroperoxides of three aldehydes isolated from cottonseed oil, viz. 2:4-decadienal, 2-octenal, and hexanal. J. W. D.

Electrolytic Process for Starch Splitting. *Chem. and Process Eng.*, **35**, 26–27 (Jan. 1954).

Starch suspension containing a small charge of NaCl is passed through a pre-heater to an electrolytic cell to which controlled quantities of acid may be added if required. After hydrolysis, which requires 10–16 min. according to the desired degree of breakdown, the soln. flows into a water-cooled neutralisation tank. The starch is separated either by centrifuging or by decantation, the electrolysing soln. returning to the mixing tank for further use. Control of time, temp., and pH is automatic, and skilled personnel is not essential. It is claimed that the process, invented by A. Hering A.G. of Nuremberg, ensures rapid, equal, and homogeneous hydrolysis to yield soln. of closely controlled and pre-determined viscosity. There is a flow diagram of the plant. J. W. D.

Stability of Pure Hydrogen Peroxide. E. M. Roth and E. S. Shanley. *Ind. Eng. Chem.*, **45**, 2343–2349 (Oct. 1953).

The Warburg manometer has been adapted to measuring the decomposition rate of hydrogen peroxide and found

to be sufficiently sensitive to provide results within a reasonable time at ordinary temperatures. Highly concentrated (>99%) H_2O_2 decomposes very slowly— approx. 0·5–1·0% per year at 30°c.—similar results being obtained in borosilicate glass and aluminium containers. Conventional stabilisers, e.g. sodium stannate, sodium pyrophosphate, 8-quinolinol do not improve the stability of pure H_2O_2, and acids decrease its stability. The decomposition rate increases approx. 2·4 times for each 10°c. rise in temp. Decomposition is chiefly a heterogeneous reaction on the walls of the container. W. K. R.

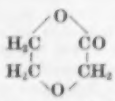
Viscosity Changes During Sol–Gel Transformation in Soap–Nujol System. K. D. Deshpande. *J. Indian Chem. Soc.*, **30**, 545–547 (Aug. 1953).

At ca. 200°F. the soap forms a mol. soln. with practically no association. The variations in viscosity with temp. exhibited during the sol–gel transformation which occurs on cooling are initially due to the passage of the sodium oleate soap soln. in Nujol to a colloidal soln. at 190°F. Subsequent changes are due to gel formation which results from (i) the gradual increase in the number and size of the soap micelles, (ii) the solvation of the micelles with the dispersion medium, which is a saturated soln. of the soap in the solvent, and (iii) the fusion of the solvated micelles into fibrils which form the gel structure enclosing the remaining liquid. The very rapid rise in viscosity during the later stages of cooling is probably due to the last of these factors. Data and graphs supporting these conclusions are presented. J. W. D.

PATENTS

Solvents for Acrylonitrile Polymers. American Cyanamid Co. *USP* 2,631,989

2-*p*-Dioxanone—



is an excellent solvent and plasticiser for acrylonitrile polymers. C. O. C.

Driers. Sherwin–Williams Co. *USP* 2,631,944

β-Methyl umbelliferone, *p*-hydroxybenzaldehyde, and *p*-hydroxypropiophenone are as good driers for unsaturated drying oils as metal soaps. C. O. C.

Carboxymethyl Cellulose in Free Acid Form (XI p. 169).

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Strengths of Some Organic Bases. K. A. Allen, J. Cymerman-Craig, and A. A. Diamantis. *J.C.S.*, 234–236 (Jan. 1954).

The basic strengths of 29 organic amines have been determined by potentiometric titration in 50% aq. ethanol at 20°c., and the results are discussed. H. H. H.

Elimination of *N*-Substituents from Derivatives of 1-Aminoanthraquinone. W. Bradley and R. F. Maisey. *J.C.S.*, 247–252 (Jan. 1954).

The behaviour of the *N*-methyl, *N*-ethyl, *N*-*n*-butyl, *N*-*tert.*-butyl, *N*-benzyl, *N*-*p*-nitrobenzyl, and *NN*-dimethyl deriv. of 1-aminoanthraquinone with regard to oxidants, sulphuric acid, and alumina shows that elimination of the *N*-substituent can occur in almost every case, the mechanism of the reaction depending on the group displaced. H. H. H.

Chemistry of Indanthrone. IX— *NN*′-Dihydro-1:2-7:8-diphthaloylphenazine. W. Bradley, R. F. Maisey, and C. R. Thitchener. *J.C.S.*, 272–273 (Jan. 1954).

Heating 2-nitro-1:1′-dianthraquinonylamine with Na_2S or $SnCl_2$–HCl affords *NN*′-dihydro-1:2-7:8-diphthaloylphenazine, which is also given by reacting 2-amino-1:1′-dianthraquinonylamine with KOH in pyridine–methanol; NaOH gives an intermediate compound. The cyclisation of 2-nitro-1:2′-dianthraquinonylamine to *NN*′-dihydro-1:2-6:7-diphthaloylphenazine is described. H. H. H.

Reduction of Anthraquinone Derivatives with Alkaline Dithionite. W. Bradley and R. F. Maisey. *J.C.S.*, 274–278 (Jan. 1954).

Extension of the work of Grandmougin and of Battegay and Hueber shows that when excess of alkaline dithionite is employed, the nature of the reduction product from anthraquinone deriv. is influenced by the kind and number of nuclear substituents, e.g. 1-piperidino- and 1-dimethylamino-anthraquinone afford deriv. of anthraquinol; 1-amino-, 1-methylamino-, 1-benzylamino-, 1-hydroxy-, 2-amino-, 2-ethylamino-, 2-dimethylamino-, and 1:5-diamino-anthraquinone give deriv. of anthrone. Generally only one form is readily isolable, even when two isomeric anthrones are theoretically possible, and, except in the case of 1-hydroxyanthraquinone, it is the form having the substituent *ortho* or *para* to a carbonyl group. 2-Nitro-1:2′-dianthraquinonylamine undergoes fission with formation of 2-aminoanthr-10-one. H. H. H.

Purpurogallin. XI— Some Further Rearrangement Reactions of the Tropolones. P. Akroyd, R. D. Haworth, and P. R. Jefferies. *J.C.S.*, 286–294 (Jan. 1954).

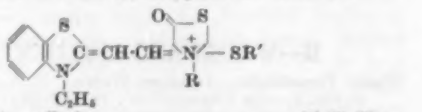
Reactions of β-methyltropolone methyl ethers with amines, alkoxides, $LiAlH_4$, and Grignard reagents are reported. The mechanisms of the reactions are discussed, and examples of the conversion of tropolones into benzenoid compounds are reported. H. H. H.

Photochromism and Reversible Multiple Internal Transitions in some *spiro*Pyrans at Low Temperatures I. Y. Hirshberg and E. Fischer. *J.C.S.*, 297–303 (Jan. 1954).

Solutions of seven *spiro*pyrans, when irradiated with ultraviolet radiation at about −100°c., afford intensely coloured modifications which are spectroscopically identical with those developed on heating ("thermochroism"). Reversion to the colourless form can also be effected by irradiation of the dye with yellow light. Ultra-violet irradiation at ~ −170°c. gives dyes which pass into those described above at somewhat higher, closely defined temp. Several of the compounds give three distinct coloured modifications. An apparatus is described for spectrophotometric determinations at any temp. between ± 180°c. H. H. H.

Some Reactions of 5-Oxothiazolinium *mero*Cyanine Dyes. R. A. Jeffreys. *J.C.S.*, 389–395 (Jan. 1954).

Several reactions, in basic media, of 5-oxothiazolinium *mero*-cyanines, of which (I) is typical, are described.



(R and R′ = alkyl). H. H. H.

Reaction of Diazonium Salts with 1-Aryl-Δ^2-pyrazolines. C. F. Duffin and J. D. Kendall. *J.C.S.*, 408–415 (Jan. 1954).

1-Aryl-Δ^2-pyrazolines (I; R = H), unsubstituted in the 3-position, react with aryl-diazonium chlorides in acetic acid to give 1-aryl-3-arylazo-Δ^2-pyrazolines (II). In alkaline solution the main products are 1:3-diaryl-Δ^2-pyrazolines (I; R = Ar). 3-Methyl-1-phenyl- and 1:3-diphenyl-Δ^2-pyrazolines with benzenediazonium-chloride, in acid solution only, give 1-*p*-phenylazophenyl deriv.

(I) (II) H. H. H.

Absence of Rearrangement in the Conversion of Benzene-*n*-diazoate into the *iso*-Diazoate, demonstrated by the use of Isotopic Nitrogen. C. A. Swan and (in part) P. Kelly. *J.C.S.*, 416–417 (Jan. 1954).

Tracer technique has indicated that in the *n*- and the *iso*-diazoate the same N atom is attached directly to the benzene nucleus. H. H. H.

Rearrangement of *N*-Acetylhydrazobenzene. D. W. Davies and D. Ll. Hammick. *J.C.S.*, 475 (Jan. 1954).

The rearrangement of *N*-acetylhydrazobenzene in conc. HCl gives *N*-acetylbenzidine and no other products, whereas in dil. HCl, and in a soln. of HCl in 95% ethanol, the products are benzidine and diphenyline in aq. acid > 5 N., and in ethanolic acid of any strength, no *N*-acetylbenzidine being detectable. The latter result is readily explained as being due to hydrolysis of the acetyl group, followed by rearrangement. H. H. H.

o-Mercapto-azo Compounds. IV—Preparation and Debenzylation of 1-(2-Benzylthiophenylazo)-2-naphthol and 1-(1-Benzylthio-2-naphthylazo)-2-naphthol. A. Burawoy, C. Turner, W. I. Hyslop, and P. Raymakers. *J.C.S.*, 82–90 (Jan. 1954).

The diazonium salt obtained either by diazotisation of 1-thiocyanato-2-naphthylamine, or by the addition of KCNS to diazotised 1-nitro-2-naphthylamine, couples with phenol in aq. Na_2CO_3 to give 2-*p*-hydroxyphenylazo-1-phenoxynaphthalene in 84% yield, and this azo- compound is also afforded by diazotised 1-nitro-2-naphthylamine; this is a novel type of replacement reaction. In like manner, *p*-cresol affords 2-(2-hydroxy-5-methylphenylazo)-1-*p*-tolyloxynaphthalene, while 1-naphthol gives the cyclic isomer of 4-(1-thiocyanato-2-naphthylazo)-1-naphthol. The anomalous courses of these reactions are discussed. Diazonium salts from *o*-aminophenyl benzyl sulphide and 2-amino-1-naphthyl benzyl sulphide are found to be stable only in conc. H_2SO_4, and a direct addition to alkaline 2-naphthol yield 1-(2-benzylthiophenylazo)- and 1-(1-benzylthio-2-naphthylazo)-2-naphthol respectively, which are debenzylated smoothly by Al Br_3 in benzene to give the corresponding *o*-mercapto-azo-compounds (~ 100% yield). H. H. H.

o-Mercapto-azo Compounds. V—Preparation and Debenzylation of 2-Benzylthio-azobenzene. A. Burawoy and C. E. Vellins. *J.C.S.*, 90–95 (Jan. 1954).

2-Benzylthio-azobenzene (I) is converted quant. by Br in glacial acetic acid into azobenzene-2-sulphonyl bromide-(II), which is also obtained, in addition to 3-benzylthiobenzidine dihydrobromide, by the action of HBr. The course of the latter reaction is elucidated. II is converted by alkali into sodium azobenzene-2-sulphinate and di-(*o*-phenylazophenyl) disulphide. The action of Al Br_3 on I is complex. H. H. H.

Colouring Matters of the Aphididæ. VIII—Studies on the Nature of the Aromatic Ring System in the Erythroaphins. B. R. Brown, A. W. Johnson, J. R. Quayle, and A. R. Todd. *J.C.S.*, 107–112 (Jan. 1954).

Fusion of the deep red erythroaphin-*sl* with Zn dust yields a complex mixture of hydrocarbons containing perylene, 1:12-benzoperylene, and coronene deriv. The nature of the carbon skeleton of the erythroaphins is discussed in the light of this evidence, and that of the similarity between the absorption spectrum of perylene and of tetra-acetyldihydroerythroaphin. H. H. H.

Infrared Absorptions and Molecular Structures of Phenol, Phenolphthalein, Fluorescein, and some Alkali Derivatives. M. Davies and R. L. Jones. *J.C.S.*, 120–125 (Jan. 1954).

The infrared data now obtained show that the "classical" representation for fluorescein is to be preferred to alternative structures, and provide definite indications of the nature of the bivalent ions and alkali addition compounds of the phthaleins referred to in the title. H. H. H.

Relation between Configuration and Conjugation in Diphenyl Derivates. III—The Ultraviolet Absorption Spectra of some 2:2′-Bridged Compounds with *meta*-Substituents. G. H. Beaven, D. M. Hall, M. S. Lesslie, E. E. Turner, and (in part) G. R. Bird. *J.C.S.*, 131–137 (Jan. 1954).

The ultra-violet absorption spectra of some substituted diphenyls bridged in 2:2′-position by the $-CH_2-CH_2-$, $-CH_2-O-CH_2-$, and

$$-CH_2-\overset{+}{N}(\diagup\!\!\diagdown)-CH_2-$$

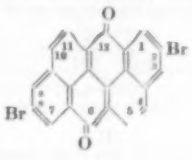
groups respectively, indicate that *mm*′-OCH_3 groups reduce the conjugation across the 1:1′-bond by mesomeric interaction with the separate benzene rings to which they are attached. This finding supports a previous view (cf. *J.C.S.*, 854 (1952)) that in non-coplanar diphenyls of this type there is still appreciable conjugation which is reduced by the introduction of either *oo*′- or *mm*′-OCH_3 groups; even in the *oo*′- case the effect of these substituents is not primarily steric. The spectrum of 2:2′-ditolyl indicates weak conjugation despite some steric hindrance to free rotation. H. H. H.

Chemistry of Anthanthrone. I—The Direct Replacement of Hydrogen by Hydroxyl and Disubstituted Amino Groups. W. Bradley and J. Waller. *J.C.S.*, 3778–3782 (Dec. 1953).

Anthanthrone (I) when heated with KOH alone or with oxidants (e.g. MnO_2, or $KClO_3$ + $CuCl_2$) is hydroxylated to the 3:9-dihydroxy derivative (II), which methylates to the 3:9-dimethoxy derivative. Morpholine, $NaNH_2$, and I afford 3:9-dimorpholino-anthanthrone, which hydrolyses to II and morpholine. The orientation of the entering groups follows the usual *op*-rule, but the absence of substitution *ortho* to the CO groups is noteworthy. This additional example of wholly *para*-substitution emphasises the remarkable character of the direct hydroxylation of anthraquinone to alizarin, in which one OH group enters the nucleus *para* and the other *ortho* to a CO group. H. H. H.

Chemistry of Anthanthrone. II—Rearrangement and Elimination Reactions in the Action of Potassium Hydroxide on Two Dibromoanthanthrones. W. Bradley and J. Waller. *J.C.S.*, 3783–3786 (Dec. 1953)

Direct bromination of anthanthrone affords the 4:10-derivative (I), identical with the compound derived by ring synthesis. Morpholine, but not aniline, replaces the Br in 2:8- and 4:10-dibromoanthanthrone, and KOH converts both into 3:9-dihydroxy anthanthrone with formation also of some anthanthrone and 4:10-dihydroxy-anthanthrone. The mechanism of the replacement, elimination, and rearrangement reactions is discussed.



H. H. H.

Carotenoids and Related Compounds. IV—A New Synthesis of the Capsorubin Chromophore. R. Ahmad and B. C. L. Weedon. *J.C.S.*, 3815–3817 (Dec. 1953).

The nonaene diketone (I), which possesses the same chromophoric system as the natural carotenoid capsorubin, is synthesised by condensation of 2:7-dimethylocta-2:4:6-triene-1:8-dial and tiglylidenepinacoline.

$[C(CH_3)_2\text{-}CO\text{-}CH{:}CH\text{-}C(CH_3){:}CH\text{-}CH{:}CH\text{-}C(CH_3){:}CH\text{-}CH{:}]_2$

(I)

H. H. H.

Interaction of Polynitro Compounds with Aromatic Hydrocarbons and Bases. XI—A New Method for determining the Association Constants for Certain Interactions between Nitro Compounds and Bases in Solution. R. Foster, D. Ll. Hammick, and A. A. Wardley. *J.C.S.*, 3817–3820 (Dec. 1953).

A new relation between the optical density and the association constants of the coloured products of the reaction in solution between nitro compounds and bases is developed, which is limited, however, to cases where the absorption of the interactants does not significantly overlap that of the products. The value of *n* in the formula AB_n for the product of interaction is readily obtained, and a check is made on the validity of the relation by a comparison of *n* for the interaction between *s*-trinitrobenzene and (1) diphenylamine, (2) dimethylamine. The compositions of these complexes, 1:1 and 1:4, respectively are confirmed by the method of continuous variations. H. H. H.

Some Properties and Reactions of Quinoline-5:8-quinones. R. Long and K. Schofield. *J.C.S.*, 3919–3924 (Dec. 1953).

6- and 7-Alkylquinoline-5:8-quinones give epoxides with H_2O_2 which are converted by conc. H_2SO_4 into 6-alkyl-7-hydroxy- and 7-alkyl-6-hydroxyquinoline-5:8-quinones respectively. Treating the quinone with conc. HCl gives a mixture of chlorides from which 6(or 7) chloro-5:8-dihydroxyquinoline may be isolated. Br_2 in CCl_4 converts 7-methylquinoline-5:8-quinone into a highly crystalline dibromide. With aniline the same quinone forms both of the possible anilinoquinones. The ultra-violet light extinction curves are discussed.

H. H. H.

Chemistry of Extractives from Hardwoods. XIV—Constitution of Jacareubin, a Pyranoxanthone from *Calophyllum brasiliense*. F. E. King, T. J. King, and L. C. Manning. *J.C.S.*, 3932–3937 (Dec. 1953).

A yellow colouring matter, jacareubin, $C_{16}H_{14}O_6$, extracted from the wood of *Calophyllum brasiliense*, contains a reactive double bond, and its trimethyl ether oxidises to a dicarboxylic acid, $C_{21}H_{20}O_{10}$, which hydrolyses with boiling HBr to give *α*-hydroxy*iso*butyric acid. Simultaneously, the nucleus is demethylated and decarboxylated to give a tetrahydric phenol, $C_{12}H_8O_6$, also obtained directly from jacareubin by alkali fusion, and identified by synthesis as 1:3:5:6-tetrahydroxy-xanthone. A pyranoxanthone structure is thus attributable to the natural product, which is regarded as 1:5:6-trihydroxy-2′:2′-dimethyl-pyrano (5′:6′-2:3 or -4:3) xanthone.

H. H. H.

Mechanism of the Coupling of Diazonium Salts with Heterocyclic Compounds. I—Glyoxaline. R. D. Brown, H. C. Duffin, J. C. Maynard, and J. H. Ridd. *J.C.S.*, 3937–3939 (Dec. 1953).

A kinetic study of the coupling of diazotised sulphanilic acid with glyoxaline (I), indicates that the rate-determining step is the reaction between the diazonium ion and the anion of I formed by ionisation of the N–H bond.

(I)

H. H. H.

Action of Hydrogen Sulphide on Certain Aromatic Amines in the presence of Formaldehyde. D. Collins and J. Graymore. *J.C.S.*, 4089–4090 (Dec. 1953).

Aniline and *p*-toluidine condense with H_2S and H·CHO to give tetrahydrothiadiazines, thia-azetidines, and dihydrodithiazines. *m*-Toluidine gave 3-*m*-tolyl-1-thia-3-azetidine, and *o*-toluidine gave an oil. All the cyclic products are rapidly decomposed by hot HCl to trithio-formaldehyde and the amine hydrochloride. H. H. H.

Two-step Preparation of Azulene from Naphthalene or Tetralin. W. Baker, W. K. Warburton, and (in part) L. J. Breddy. *J.C.S.*, 4149–4154 (Dec. 1953).

The production of up to 0·25% of azulene by dehydrogenation of certain commercial specimens of decalin is shown to be due to the presence of decahydroazulene. The latter is found to be derived from naphthalene, *via* tetralin, by rearrangement of the C-skeleton during reduction at high temp. and pressure over a W or Mo sulphide catalyst. Azulene is most conveniently separated from mixtures by extraction with conc. HCl.

H. H. H.

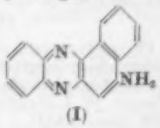
Chlorination of Arylamine Derivatives by Sulphuryl Chloride. F. Bell. *J.C.S.*, 4182 (Dec. 1953).

The very variety of compounds obtained when sulphuryl chloride reacts with aromatic compounds in the presence of various catalysts, has hitherto militated against the use of this reagent as a nuclear chlorinating agent. It is now found that by addition of sulphuryl chloride to certain compounds (of which a number are cited; e.g. 1-chloro-2-acetonaphthalide converted into 1:3:4-trichloro-2-acetonaphthalide), with subsequent removal of the excess of reagent and crystallisation of the residue, a number of derivatives, otherwise difficult of access, are obtained in high yield.

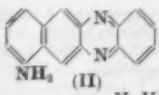
H. H. H.

Reaction of *o*-Nitroaniline with 1-Naphthylamine. J. W. Cook and L. Hunter. *J.C.S.*, 4183 (Dec. 1953).

It is shown that when *o*-nitroaniline and 1-naphthylamine are heated together with $ZnCl_2$, 3-amino-1:2-benzophenazine (I) is produced. The structure (II) formerly assigned, now becomes invalid.



(I)



(II)

H. H. H.

Preparation and Deamination of 4:4′-Diamino-3:3′-dinitrodiphenyl. C. C. Barker and F. D. Casson. *J.C.S.*, 4184–4185 (Dec. 1953).

Nitration of 4:4′-diacetamidodiphenyl in acetic anhydride–nitric acid (95%) at −12° gives 4:4′-diacetamido-3:3′-dinitrodiphenyl in 97·5% yield, which is hydrolysed to the diamine by dilute sulphuric acid at 100°. Deamination of the tetrazonium salt, prepared from the diamine and nitrosylsulphuric acid (cf. Hodgson and Walker, J., 1933, 1620), by addition to a large amount of ethanol with Cu_2O in suspension, gives a 94% yield of 3:3′-dinitrodiphenyl, which is reduced by iron pin dust and HCl in 85% ethanol to 3:3′-diamino-diphenyl (overall yield, 75%).

H. H. H.

6:7-Benzothionaphthen. W. Carruthers. *J.C.S.*, 4186–4187 (Dec. 1953).

6:7-Benzothionaphthen (I), isolated by Kruber and Raeithel (*Chem. Ber.*, **86**, 366 (1953)) from a coal-tar fraction, has now been prepared by reduction of 6:7-benzothioindoxyl with Zn dust and acetic acid, the mixture obtained being converted to the pure product by dehydrogenation with sulphur. Desulphurisation of I with Raney Ni gives 2-ethylnaphthalene, whilst oxidation of I with CrO_3 gives the 4:5-quinone.

H. H. H.

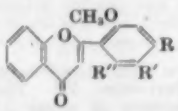
2:3- and 2:5-Diaminoquinol Diethyl Ether. E. S. Lane and C. Williams. *J.C.S.*, 4187–4188 (Dec. 1953).

The above ethers, required as intermediates for the preparation of new metal-complexing agents, are prepared by catalytic hydrogenation of the nitration product of quinol diethyl ether. Attention is drawn to the ease of separation of the isomers based on the difference in solubility of the hydrochlorides in ethanol, and to the extreme ease with which the 2:5-isomer undergoes atmospheric oxidation compared with the stability of the 2:3-isomer.

H. H. H.

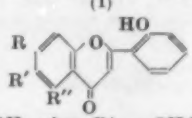
Rearrangement in the Demethylation of 2′-Methoxyflavones. K. M. Gallagher, A. C. Hughes, M. O'Donnell, E. M. Philbin, and T. S. Wheeler. *J.C.S.*, 3770–3777 (Dec. 1953).

Certain 2′-methoxyflavones (I) rearrange during demethylation by HI under sufficiently drastic conditions, to give the related 2′-hydroxyflavones (II) in which the 2-phenyl group and the fused aromatic ring of the original flavone are interchanged. Demethylation by $AlCl_3$ does not produce rearrangement. The more stable of each pair of related 2′-hydroxyflavones, is that in which the 2-phenyl side chain contains the smaller number of OH groups.



(R = H,OCH_3,H,OCH_3, when R′ = OCH_3,H,H,H, and R″ = H,H,OCH_3,OCH_3, respectively).

(I)



(R = H,OH,H,OH, when R′ = OH,H,H,H, and R″ = H,H,OH,OH, respectively).

H. H. H.

(II)

Flavanoid Biogenesis in *Chlamydomonas Eugametos*. A. J. Birch, F. W. Donovan and F. Moewus. *Nature*, **172**, 902–904 (14 Nov. 1953).

Genetics of flavonoid pigment production are studied using the green alga *Chlamydomonas Eugametos* which uses the flavenoid isorhamnetin and the anthocyanin peonin as sex-hormones. J. W. B.

Effect of Melanophore Hormone on Regeneration of Visual Purple in Solution. T. Hanaoka. *Nature*, **172**, 866 (7 Nov. 1953).

Visual purple obtained from frogs is bleached by light and re-incubated in the dark with and without melanophore hormone, which is found to increase regeneration of the purple. J. W. B.

Aromatic Diazo Compounds. V—Coupling of Benzene Diazonium Chloride with 4-Nitroresorcinol. J. A. Langner and Z. J. Allan. *Chem. Listy*, **46**, 247–248 (1952): *Chem. Abs.*, **47**, 8669 (10 Sept. 1953).

4-Nitroresorcinol in aq. NaOH coupled with diazotised aniline to give mainly 2:6-bisphenylazo-4-nitroresorcinol with some 2-phenylazo-4-nitroresorcinol and a trace of 4-phenylazoresorcinol. The amino- and diaminoresorcinols obtained from these azo compounds by reduction with Zn were identified by paper chromatography tests.

VI—Reaction of Diazonium Salts with Hydroquinone. J. Dobáš. *Ibid.*, 277–280: *Chem. Abs.*, **47**, 8669 (10 Sept. 1953).

Hydroquinone reacted with diazotised *o*-nitro-, *p*-nitro-, and 2:4-dinitro-aniline with loss of N_2 to give 2:5-dihydroxy-2′-nitrodiphenyl and 2:2″-dinitro-2′:5′-dihydroxy-*p*-triphenyl, 2:5-dihydroxy-4′-nitrodiphenyl, and 2:5-dihydroxy-2′-4′-dinitrodiphenyl respectively.

VII—Coupling with *m*-Phenylenediamine. F. Mužík and Z. J. Allan. *Ibid.*, 350–357: *Chem. Abs.*, **47**, 8669 (10 Sept. 1953).

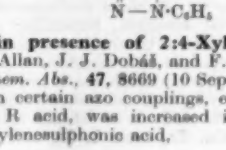
4-Phenylazo-*m*-phenylenediamine (chrysoidine) coupled with diazotised aniline to give a mixture of 2:4:6-tris-, 2:4-bis-, and 4:6-bis-(phenylazo)-*m*-phenylenediamine. The trisazo compound boiled with $CuSO_4$ in pyridine gave 2:5-diphenyl-*ang*-benzobistriazole (2:7-diphenylbenzo(1:2-3:4)-bistriazole), and the disazo compound gave 6-phenylazo-5-amino-2-phenyl-2H-benzotriazole. Similar reactions occured using, e.g., 4-phenylazo-*m*-toluylenediamine and diazotised *p*-chloraniline.

VIII—Coupling with Benzidine. Z. J. Allan and F. Mužík. *Ibid.*, **46**, 485–486: *Chem. Abs.*, **47**, 8705 (10 Sept. 1953).

Diazotised *p*-nitroaniline with benzidine gave 4-*p*-nitrobenzenediazoamino-4′-aminodiphenyl, which by boiling with aq. HCl gave *p*-nitroaniline and 4-aminodiphenyl-4′-diazonium chloride. *NNN′N′*-tetramethylbenzidine reacted with diazotised *p*-nitro- and 2:4-dinitro-aniline with elimination of N_2 to give respectively 3-*p*-nitrophenyl- and 3-*op*-dinitrophenyl-*NNN′N′*-tetramethylbenzidine.

IX—2:5:8-Triphenylbenzotristriazole. F. Mužík and Z. J. Allan. *Ibid.*, 487–488: *Chem. Abs.*, **47**, 8705 (10 Sept. 1953).

Trisphenylazo-*m*-phenylenediamine on oxidation with $CuSO_4$ gave 2:7-dihydro-2:7-diphenyl-4-phenylazobenzo (1:2:3:4) bistriazole, which yielded by hydrogenation over Raney Ni the corresponding 4-amino compound. This coupled with diazotised aniline to give the 5-phenylazo compound, which on oxidation with $CuSO_4$ in pyridine gave 2:5:8-triphenylbenzotristriazole.

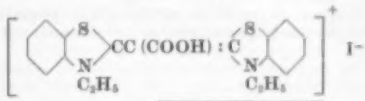


X—Coupling in presence of 2:4-Xylenesulphonic Acid. Z. J. Allan, J. J. Dobáš, and F. Mužík. *Ibid.*, 628–631: *Chem. Abs.*, **47**, 8669 (10 Sept. 1953).

The yield from certain azo couplings, e.g. 2-amino-4-chlorophenol → R acid, was increased in presence of pyridine or 2:4-xylenesulphonic acid. E. S.

Monomethincyanines. Y. Hishiki. *J. Sci. Research Inst.* (Tokyo), **45**, 112–127 (1951): *Chem. Abs.*, **48**, 65 (10 Jan. 1954).

The synthesis is described of thiacyanine periodide, thiazolocyanine periodide, oxacyanine periodide, naphthocyanine periodide, 3:3′-diethylmonomethinthiocyanine iodide, 3:3′:4:4′-tetramethylmonotriethinthiazolcyanine-iodide, 3:3′-dimethylmonomethinoxacyanine iodide, 3:3′-diethylmonomethin-*a*-naphthothiacyanine iodide, and the compounds—



and $[p\text{-}(CH_3)_2NC_6H_4CH{:}C(\overline{C{:}NCH_2{\cdot}CCH_2{:}CH{\cdot}S})_2]I_2$.

C. O. C.

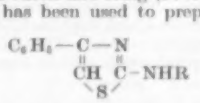
Solubility and Light Fastness of Azo Dyes—III. A. B. Sen and R. C. Sharma. *J. Indian Chem. Soc.*, **30**, 503–508 (July 1953).

Thirty new azo dyes have been prepared from *m*- and *p*-aminoacetophenones. The preparation is described, the colours listed (in the form of the Lovibond colour nomenclature) for the dry powders, and the light fastness reported. The introduction of the $-COOCH_3$ group into the amino-grouping reduces the light fastness and the solubility of the dyes in benzene and in oil.

J. W. D.

Preparation of *N*-Substituted 2-Aminothiazoles. I—Condensation of Acetophenone with Substituted Thioureas. G. N. Mahapatra and M. K. Rout. *J. Indian Chem. Soc.*, **30**, 398–400 (June 1953).

The method of Dodson and King (*J. Amer. Chem. Soc.*, **67**, 2242 (1945)) has been used to prepare—



in which R is, in turn, phenyl, *o*-, *m*-, and *p*-tolyl, *o*-, *m*-, and *p*-chlorophenyl, *o*-, *m*-, and *p*-carboxyphenyl, and *α* and *β*-naphthyl. The picrates and acetyl derivatives have also been isolated and characterised.

J. W. D.

Improved Method for the Preparation of Bis-2-thionaphthen-ethylene-indigos. S. K. Guha and J. N. Chatterjea. *J. Indian Chem. Soc.*, **30**, 379–382 (June 1953).

The preparation is described of bis-2-thionaphthen-ethylene-indigoid dyes from 3-hydroxythionapthen and its 5-methyl, 7-methyl, 5-chloro, 7-chloro, and 5-bromo-substituted derivatives. Use of glyoxal sulphate instead of glyoxal sodium bisulphite (*GP* 239,916) considerably improves the yield and shortens the reaction time. Condensation takes place readily in acetic acid soln., H_2SO_4 released *in situ* acting catalytically; in some cases the yield may however be improved by a catalytic trace of HCl. Yields > 70% are reported. J. W. D.

Ferric Complexes of Phenols. I—Phenol and *m*-Cresol. II—Salicylaldehyde and *o*-Hydroxyacetophenone. S. K. K. Jatkar and B. N. Mattoo. *J. Indian Chem. Soc.*, **30**, 592–600, 607–609, (Sept. 1953).

I—Colorimetric titration of $FeCl_3$ with phenol at M/400 Fe^{3+} shows the existence of the complex $Fe(OR)^{2+}$, which is shown by the calculation of the instability or equilibrium const. At higher concn. (M/100 Fe^{3+} and M/15 Fe^{3+}), Job's method of continuous variation (*Ann. Chim.*, **9**, 113 (1928)) shows the existence of $Fe(OR)^{2+}$ and $Fe(OR)_3$ respectively with phenol and *m*-cresol. Potentiometric studies at higher concn. using a Pt indicator electrode, show the presence of a series of complexes, $Fe(OR)_n^{n-n}$, where n = 1, 2, 3, . . . 6. The colour of the complexes is explained quant. as resulting from differences between various ultraviolet and visible absorption frequencies of the constituent chromophores.

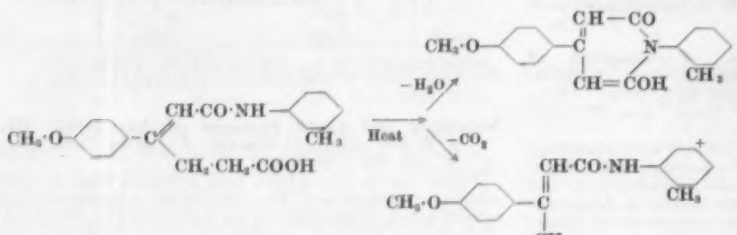
II—The method of continuous variation shows that the mono-complexes (equimolecular) are formed with both

salicylaldehyde and *o*-hydroxyacetophenone. In the colorimetric titration, a 16-fold excess of Fe^{3+} is required to secure practically complete shift of the following equilibrium to the right, giving colour saturation—

$$Fe^{3+} + [OR]^- = Fe(OR)^{2+}$$ J. W. D.

Action of Aniline and Other Primary Amines on the Anhydrides of β-Arylglutaconic Acids. I—Formation and Decarboxylation of Semianilides. S. S. Karmarkar and V. M. Bhave. *J. Indian Chem. Soc.*, **30**, 689–694 (Oct. 1953).

The thermal degradation of the *o*-methylsemianilide of β-4-methoxyphenylglutaconic acid proceeds as—



and has been found to be typical of the semianilides of β-arylglutaconic acids (numerous other examples are are given). Other previously known semianilides of β-(methoxyphenyl)-glutaconic acid behave similarly. The *p*-carbomethoxysemianilide of β-(4-methoxyphenyl)-glutaconic acid, and *o*-carbomethoxysemianilide of β-(2-methyl-4-methoxy-5-*iso*propylphenyl)-glutaconic acid are hydrolysed by NaOH to the corresponding carboxy acids, as are also the decarboxylation products of certain of the above degradations. These acids are converted by acetic anhydride to lactones which may be hydrolysed by NaOH to give the original acids. The structure of these decarboxylation products is still in some doubt. J. W. D.

Explosive Nature of the Reaction between Certain Diazotised Substituted Benzenes and Sodium Polysulphide. R. G. W. Hollingshead and P. E. Garner. *Chem. and Ind.*, 1179–1180 (31 Oct. 1953).

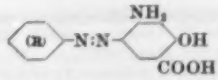
Reactions between Na_2S_x and the diazo compounds of any of the following amines lead to dangerous explosions — *o*-nitroaniline, *m*-chloroaniline, 4-chloro-*o*-toluidine, and 2-naphthylamine. The analogous reaction using diazotised anthranilic acid has, however, been in use for many years in the dyestuffs industry for the preparation of diphenyl-disulphide-2:2′-dicarboxylic acid (a sulphophthalein dye intermediate), and no explosion has been reported. Eight separate experiments in which conditions were modified to upset the balance of the latter reaction showed no sign of explosion or loss of control. Unlike the other instances cited, the diazo compound of anthranilic acid has some solubility in water, and the separation by water mol. may exert a moderating influence which is absent when the primary reaction product separates from soln. It has also been suggested that the intermediate Ar·N:N·S·S·N:N·Ar may be spontaneously explosive. A further theory is that diazonium salts containing carboxyl and/or sulphonic groups may, unlike those containing nitro and/or halogen groups, exist as zwitterions of the betaine type. J. W. D.

Some Trihalogenated Nitrobenzenes and their Derivatives—II. S. S. Joshi and V. P. Saxena. *J. Indian Chem. Soc.*, **30**, 548–550 (Aug. 1953).

Preparation of 1:2:3-tribromobenzene, 1:3-dibromo-2-chlorobenzene, 1:2:3-trichlorobenzene, and 1:3-dichloro-2-bromobenzene and the conversion of these to their 4:6-dinitro derivatives is described. All yield derivatives readily with aniline, *o*- and *p*-toluidines, diethylamine, piperidine, and ammonia; the identity, colour, and m.p. of each of these latter is given. J. W. D.

***p*-Aminosalicylic Derivatives (Dyes)—I.** S. N. Mahapatra and S. S. G. Sircar. *J. Indian Chem. Soc.*, **30**, 563–567 (Aug. 1953).

The preparation is described of azo dyes having the general structure—



(R) = Cl, NO_2, CH_3, etc. in *o*-, *m*-, or *p*-position

formed by coupling *p*-aminosalicylic acid (I) in alk. soln. with several diazotised simple aromatic amines (including all the toluidines, monochloroanilines, mononitroanilines, naphthylamines, aminobenzoic acids, and aniline and benzidine themselves). Others were prepared similarly using the methylol derivative resulting from the equimolecular condensation of (I) with HCHO. A number of amines were mercurated before coupling, thus yielding a further series of dyes. None of the dyes was found to be very toxic. The antibacterial action against *B. coli*, *Staphylococcus aureus*, and *Mycobacterium phlei* is high with the mercurated dyes, but very low with the others. J. W. D.

New Synthesis of *p*-Nitrobenzenesulphonyl Chloride. K. Raman and M. Raghavan. *J. Indian Chem. Soc.*, **30**, 538–542 (Aug. 1953).

Sulphanilic acid is diazotised, and the diazonium group is replaced by $-NO_2$ by treatment with dil. HNO_2 and Cu_2O. The resulting *p*-nitrobenzene sulphonic acid is converted to the K salt which yields *p*-nitrobenzene sulphonyl chloride on treatment with PCl_5 or $Cl{\cdot}SO_3H$. The pure compound is obtained directly at the end of the reaction sequence, and the tedious and difficult purifications associated with other methods of synthesis are obviated. Yields of 27–59% are obtained, according to the reaction conditions. J. W. D.

Pechmann Condensation. I—Condensation of 2:5-Disubstituted Phenols with Ethyl Acetoacetate. R. N. Usgaonkar, V. M. Thakor, and G. V. Jadhav. *J. Indian Chem. Soc.*, **30**, 535–538 (Aug. 1953).

2-Ethyl- and 2-propyl-5-methoxyphenols on condensation with ethyl acetoacetate in presence of $AlCl_3$ or H_2SO_4 yield 6-ethyl- and 6-propyl-7-methoxycoumarin respectively, the alkyl groups having migrated. When the condensation of the former is effected in presence of P_2O_5, 5-methoxy-4-methyl-8-ethylcoumarin is obtained. 2-Ethyl-5-methylresorcinol similarly yields 7-hydroxy-4:5-dimethyl-8-ethylcoumarin, in presence of H_2SO_4, $AlCl_3$, or P_2O_5. J. W. D.

Cadmium Pigments. P. J. Curtis and R. B. Wright. *J. Oil and Col. Chem. Assocn.*, **37**, 26–43 (Jan. 1954).

Cadmium pigments have good resistance to heat and to chemical attack, and are of good colouring power [*sic*]; since Cd is a by-product of Zn smelting, output largely governs price. Cadmium yellows consist of the sulphide and are manufactured by pptn. (the conditions of which regulate the colour and pigmentary quality) followed by drying and calcining; this, and other alternative methods of manufacture are discussed briefly. The various theories advanced to explain the shade differences amongst Cd yellows are reviewed; this is still open to controversy. In most modern methods, Cd–Se reds are made by the co-pptn. of Cd sulphide and selenide followed by calcination; they are believed to consist of solid soln. of Cd sulphide and selenide crystals. Yellow lithopones may be made by the double pptn. of CdS and $BaSO_4$ followed by calcination. Green-toned yellows result if some of the Cd is replaced by Zn in the pptn., and typical compositions of such lithopones are given. Because of their good pigmentary properties and economy in use, the lithopones are growing in importance. Red lithopones are similarly made by introducing Se into the pptn. mixture. Simple mechanical extensions of Cd pigments are unsatisfactory, being of inferior tinctorial strength, durability and pigmentary quality; the extender is inclined to separate out when such mixtures are dispersed in a medium.

Qualitative and quantitative tests are described whereby Cd pigments may be examined for extenders and adulterants. The quant. analysis of Cd and total S in CdS pigments, and of Cd, Zn, total S, Se, and insolubles in Cd

yellow lithopones, extended pigments, Cd selenosulphide pure pigments and lithopones, is detailed. Physical properties discussed under separate headings, with graphs where required, are — trichromatic loci, sp.gr., particle size, oil absorption, stability in paint media, and toxicity. The excellent light-fastness of Cd pigments is reduced by the presence of TiO_2. The uses of Cd pigments in various industries are discussed under headings which include— paint, artists' colours, rubber, plastics (especially P.V.C.), printing inks, wallpaper, and leather finishes.

J. W. D.

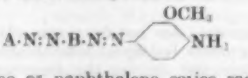
PATENTS

Dischargeable Dis- and Poly-Azo Direct Cotton Dyes. S. *BP* 701,709

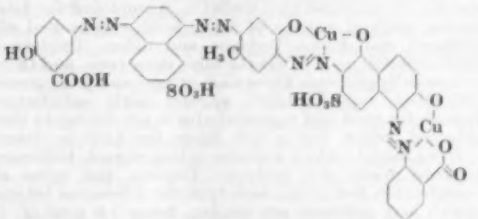
Two mol. of an amino-mono-, -dis-, or -poly-azo compound free from groupings capable of metal-complex formation, but containing at least two SO_3H and/or COOH groups, are condensed with 1 mol. of a dibasic acid dihalide X·CO·CH:C(A)·CO·X (A = H or CH_3; X = Cl or Br) to give dischargeable yellow, orange and brown direct cotton dyes. Thus to a soln. in aq. NaOH of the disazo compound 4-aminoazobenzene-3:4′-disulphonic acid → *m*-toluidine are added slowly a benzene soln. of fumaryl dichloride and sufficient aq. Na_2CO_3 to maintain a weekly alkaline reaction throughout. The product dyes cotton a dischargeable yellow-orange. E. S.

Grey, Copperable, Tetrakisazo Direct Cotton Dyes. S. *BP* 703,133

2:5-Dihydroxynaphthalene-7-sulphonic acid is coupled with 1 mol. of an arylamine having in the *ortho* position a group capable of metal-complex formation and 1 mol. of a diazotised aminodisazo compound—



(A = a benzene or naphthalene series radical; B = a benzene or naphthalene series radical in which the azo groups are *para* to one another; CH_3, OCH_3 and acylamino groups may be present) to give tetrakisazo dyes which may be coppered in substance or on the fibre. The components are so chosen that the final dye contains > 1 SO_3H group. The same dyes may be made by first coupling one of the diazo compounds with 2-amino-5-naphthol-7-sulphonic acid and replacing the 2-amino group by OH before carrying out the second coupling. Coppering may also be carried out after the first coupling, and the copper complex so formed is then coupled with the second diazo compound. Thus, the 2-amino group of the copper-complex of the monoazo compound anthranilic acid→ 2-amino-5-naphthol-7-sulphonic acid is replaced by OH, and the compound so formed is coupled in aq. ammoniacal pyridine with the diazo compound of the aminodisazo dye 5-aminosalicyclic acid → 1-naphthylamine-7-sulphonic acid → *p*-cresidine. The product dyes cellulosic fibres grey by the after-coppering process, or it may be converted in substance to the copper-complex—



by heating with ammonical copper sulphate so that the CH_3 is removed from the OCH_3 group of the cresidine residue. E. S.

Alkylaminoanthraquinones. S. *BP* 700,044

Aliphatic amines with 4-8C atoms are condensed with anthraquinone compounds carrying replaceable substituents, e.g. Hal, OH, OAlk, NO_2 or SO_3H in the 1- and 5- or 1- and 8-positions to give red colouring matters soluble in oils, fats, and organic solvents. Thus 1:5-dichloroanthraquinone is heated at 90–95°C. for 16 hrs. in 1-aminopentane in presence of $CuSO_4$ and Na_2CO_3.

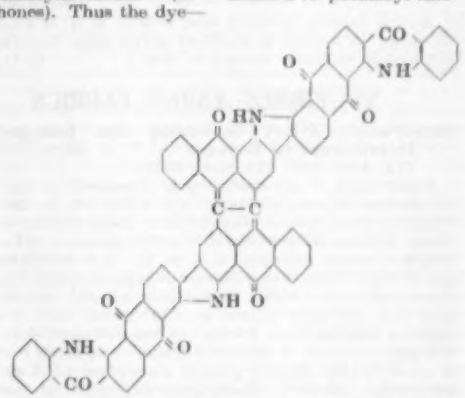
R. K. F.

Brominated 1-Amino-4-arylaminoanthraquinone-2-sulphonic Acids— Acid Dyes. S. *BP* 699,781

Dyes obtained by condensing the alkali metal salts of 1-amino-4-bromoanthraquinone-2-sulphonic acid with a 5:6:7:8-tetrahydronaphthylamine or a *p*-aminobenzanilide are brominated to give blue neutral dyeing acid dyes of good fastness to milling and perspiration. Thus 1-amino-4-(5′:6′:7′:8′-naphthyl-1′-amino)-anthraquinone-2-sulphonic acid is treated in H_2SO_4 at 20–60°C. with a solution of bromine in H_2SO_4. R. K. F.

Acedianthrone Vat Dyes. CFM. *BP* 699,149

Grey, brown and olive green vat dyes are obtained by cyclising to the corresponding carbazoles, the condensation products of halogenoacedianthrones and amino-3′-4′-phthaloylacridones (or amino-3′:4′-phthaloyl-thioxanthones). Thus the dye—



is made by heating the dichloroacedianthrone obtained from 1-chloroanthrone, with 3′:4′-phthaloyl-4-aminoacridone in nitrobenzene at 200–205°C. in presence of sodium acetate and cupric acetate. The resulting anthrimide is heated at 70–75°C. in a mixture of $AlCl_3$ and NaCl through which SO_2 is passed. R. K. F.

4′:4″-Bis(2:4-dialkoxybenzoylamino)-stilbene-2′:2″-disulphonic Acids— Fluorescent Brightening Agents. American Cyanamid Co. *BP* 700,510

The amine salt or the *p*-diaryl guanidine salt of 4:4′-diaminostilbene-2:2′-disulphonic acid is treated with a 2:4-dialkoxybenzoyl halide to produce fluorescent brightening agents of good substantivity to and fastness on cellulose. Thus 4:4′-diaminostilbene-2:2′-disulphonic acid is neutralised with aq. NaOH and di-*o*-tolylguanidine as its aq. hydrochloride added. The resulting *o*-toluguanidine salt is separated and heated in nitrobenzene at 150°C. with 2:4-dimethoxybenzoyl chloride. Neutralisation and separation follow by adding a soln. of phenol in 5N-NaOH. R. K. F.

7-Bromo-5-chloroisatin. General Aniline. *BP* 699,769

7-Bromo-5-chloroisatin is made either by direct bromination of 5-chloroisatin in an inert solvent, e.g. ethanol, or by treating 1:5-dichloroisatin in a solution containing bromide ions. Thus 1:5-dichloroisatin (prepared as described in *GP* 255,772) is suspended in aq. KBr containing H_2SO_4 at 60°C. R. K. F.

Black Iron Oxide. Reconstruction Finance Corpn. *USP* 2,631,085

Yellow iron oxide, $Fe_2O_3·H_2O$, is treated in aqueous slurry with hydrated ferrous oxide $FeO·H_2O$, using 2 parts by weight of the ferric oxide to 1 part of the ferrous, to yield the black oxide $Fe_2O_3·FeO$. The pH of the mixture should be kept at < 7 and the reaction is complete in 2 hr. at 200°F. The best black is obtained if a current of air is passed through the slurry during the reaction.

C. O. C.

New Method for Preparing Dimethyl Terephthalate from Toluene— A Contribution to the Problem of Raw Materials for Synthetic Fibres (VI p. 165).
Photographic Colour Development. I—Rate of Colour Development (IX p. 167).
Leather Dyes— a Survey (XII p. 170).

V—PAINTS; ENAMELS; INKS

Temperature-indicating Paints. J. E. Cowling, P. King, and A. L. Alexander. *Ind. Eng. Chem.*, **45**, 2317–2320 (Oct. 1953).

A series of compounds is listed, which when incorporated into a suitable vehicle, preferably a plasticised methacrylate resin solution, and applied as paints, produce characteristic colour changes at specific temps. within ± 5°C. The selected compounds do not show rapid reversibility or give similar changes after prolonged heating at lower temps., and cover the range 50–270°C. W. K. R.

PATENT

Steam-setting Printing Inks. American Cyanamid Co. *USP* 2,631,986

An ink capable of being used at high R.H. is produced by using as vehicle a modified alkyd resin dissolved in a dihydric aliphatic alcohol of 4–6 C. C. O. C.

VI— FIBRES; YARNS; FABRICS

Small-angle X-Ray Scattering and Interparticle Interference in Rayons. A. N. J. Heyn. *Nature*, **172**, 1000–1001 (28 Nov. 1953).

Small-angle X-ray scattering is measured on two high-orientation rayons (Fortisan and Fiber G), in the dry, water-swollen, and swollen beyond water-swollen conditions. Examination of the diffraction patterns and angle–density curves indicates that in the dry condition the scattering is limited to a narrow angular distance from the centre, whereas swollen fibres show a wider distribution and also inflexion points in the curve, with distinct maxima and minima. Fibres swollen still more show only a slight inflexion, or none at all, and the overall intensity is much higher despite greater absorption of X-rays by the swollen material. Results are explained on the basis of interparticle interference in the denser states of packing, and particle sizes of 45·9A. for Fortisan and 40·6A. for Fiber G are calculated. J. W. B.

Resistant Membranes from Wool and Hair Fibres. B. Manogue and M. S. Moss. *Nature*, **172**, 806–807 (31 Oct. 1953).

Transverse sections of wool and hair fibres which have been treated with peracetic acid and embedded in ester wax are cut to 0·4–2·0μ. The ribbons are transferred to water containing a little ethoxy-ethyl alcohol on a slide, dried at 35°C., and the wax removed with hot xylene. The sections are treated 5–10 min. with 2N-NaOH, washed, dried, and flooded with 0·3% nitrocellulose in amyl acetate. They can then be stripped off on the nitrocellulose support and examined. It is seen that the boundaries of the cortical cells, cell nuclei, and some fibrils are resistant to the treatment; the membranes surrounding the cortical cells are about 100A. thick, about the same as the epicuticle. J. W. B.

Polyethylene Terephthalate Fibres— Fine Structure. A. Cobbold, R. de P. Daubeny, K. Deutsch, and P. Markey. *Nature*, **172**, 806 (31 Oct. 1953).

Working with monofilaments (0·25 mm. and above) a reproducible technique is devised for exposing the internal structure of polyethylene terephthalate fibres. The monofil is simply torn down its length, the structure apparently guiding the tear along planes of comparatively low cohesive strength, thus revealing rather than obliterating the fine details. Replicas are produced by the evaporation technique, using germanium as the replicating medium, and electronmicrographs show clearly the essentially fibrillar character of the fine structure. Quantitative evaluation is only approximate but suggests that 200-A. diameter fibrils might be fundamental, grouping together to a maximum of about 1000 A., the aggregate appearing as a larger fibril. J. W. B.

Decrystallised Cotton. G. Susich. *Amer. Dyestuff Rep.*, **42**, 713–718 (26 Oct. 1953).

When cotton, as staple fibre or yarn, is treated in liquid anhydrous ethylamine, preferably at ice-bath temperature, the amine removed by extraction with chloroform or hexane, and dried, the amine acts as a swelling agent and reduces fibre crystallinity. Propylene, and higher amines preceded by ethyl- or methylamine, reduce crystallinity by controllable degrees. Modifications in structure as shown by X-ray data are discussed, and an account is presented of tests carried out on ordinary and decrystallised cotton to determine the comparative tensile, abrasive, knotting, and wet-dry properties. Extensibility and energy for rupture are increased, and wet recovery from extension improved, without sacrificing other advantages. Good knot and wet behaviour is the result of unchanged fibrillar structure, unchanged cellulose I lattice, and high degree of polymerisation. It has a disadvantageous low initial modulus and high permanent set; abrasive damage is higher and similar to that of staple viscose. It is suggested that by controlling conditions during swelling, or by cross-linking, the properties may be so modified to suit the cotton to purposes for which it has hitherto been unsatisfactory. J. W. B.

Cotton— An Old Fibre with a New Future. L. Smith. *Amer. Dyestuff Rep.*, **42**, P789–P791 (23 Nov. 1953).

A survey of cellulose reactions which have applications in cotton modification, and the production of speciality fibres and special finishes thereby. J. W. B.

Classification of Textile Fibres. *J. Textile Inst.*, **44**, s 69–s 70 (Dec. 1953).

A table is presented in which textile fibres are classified under main headings of natural and man-made, natural fibres being subdivided as animal, vegetable and mineral, and man-made being subdivided as natural polymers, synthetic polymers, and non-polymeric. J. W. B.

Structure of Flax Fibre Strands. D. R. Gilling and D. E. J. Samuels. *J. Textile Inst.*, **44**, T 609–T 618 (Dec. 1953).

If short segments are cut from strands of hackled line flax, many segments fall apart into smaller pieces or even single ultimates; the shorter the segment the larger the number of pieces. This leads to a new conception of a retted fibre strand as a unit in which the binding materials between the ultimate fibres are interrupted by a succession of longitudinal fissures. The system of fissuring is characterised by the number-length curve when a tuft of fibre strands is cut into short segments, and may have an important bearing on the mechanism of splitting down the fibre strands during processing. A method is described for determining the number-length curve, and results obtained are analysed and a theoretical model of strand structure calculated. Factors likely to be causally related to the system of fissuring are the degree of retting, in which binding material is disrupted; storage, as long-stored samples show more fissuring; and varietal differences, as different varieties have different types of fibre bundling which may influence fissuring. J. W. B.

Heat of Wetting of Fibres. N. F. H. Bright, T. Carson, and G. M. Duff. *J. Textile Inst.*, **44**, T 587–T 595 (Dec. 1953).

A constant-heat-loss calorimeter in which temperature rise can be measured to ±0·0001°C. is employed for determining heats of wetting of wool, Ardil, regenerated silk, undrawn and drawn Terylene and nylon, Terylene of three different deniers but the same draw ratio, and Orlon. Although hygroscopic fibres such as wool and Ardil present difficulties, reproducibility appears fairly satisfactory. Results for wool and regenerated silk are similar to those obtained earlier, but a new figure for Ardil is claimed —26·9 g.cal./g.—which is similar to that of wool. Differences between drawn and undrawn Terylene and nylon are considerable, but within each type the differences between drawn and undrawn are similar, being 1·0 g.cal./g. for nylon and 0·6 g.cal./g. for Terylene. If heats of wetting of specially drawn Terylene are plotted against the square root of the denier the points lie nearly on a straight line passing through the origin, implying that the heat of wetting of drawn Terylene is proportional to the external surface area, at 4×10^{-4} g.cal./sq.cm. Other drawn and undrawn results do not fall on the same graph, which is difficult to explain, though it may mean that the surface available for sorption is considerably greater than the external surface, the ratio of the surfaces only being constant for constant draw ratio. When differential heats of wetting are plotted against R.H., Ardil gives a different graph from wool and nylon, and it is suggested that the

fibre swells greatly during initial sorption, thus leading to the heat liberated being utilised in expanding the lattice, and a lower figure for effective heat of wetting.
J. W. B.

New Method for Preparing Dimethyl Terephthalate from Toluene—A Contribution to the Problem of Raw Materials for Synthetic Fibres. W. Griehl. *Faserforsch. und Textiltech.*, **4**, 464–469 (Nov. 1953).

In a preparation of terephthalic acid, toluene is converted into *o*- and *p*-chloromethyltoluenes by treatment with H·CHO and HCl in presence of $ZnCl_2$, and the mixture obtained, after purification, is side-chain chlorinated, yielding a mixture of ω-pentachloro-*o*-xylene and ω-hexachloro-*p*-xylene, which react quantitatively with water at 120–140° in presence of $FeCl_3$ to give a mixture of phthalaldehydic acid and terephthalic acid, readily separable by treatment with water. Dimethyl terephthalate is prepared by the action of aq. methanol, in presence of an acid and an Fe or Zn salt, on ω-hexachloro-*p*-xylene, which is separated from the accompanying ω-pentachloro-*o*-xylene (in the mixture prepared as already described) by fractional crystallisation from light petroleum (the portion remaining in soln. being converted into terephthalic acid as already described). Utilisation of by-products, particularly phthalaldehydic acid and methyl chloride, and other matters of importance in large-scale running are considered.
A. E. S.

Relation between Lustre, Brightness, and Surface Characteristics of Textiles. O. De Riz. *Faserforsch. und Textiltech.*, **4**, 472–476 (Nov. 1953).

Lustre and brightness ("white-content") measurements on a series of patterns of graded brightness prepared from matt rayon-staple sliver, undyed and dyed with various amounts of Sulphur Black, show that there is a linear relationship between the logarithms of these quantities. Measurements are made also on yarn and fabrics prepared from the slivers, and these also yield linear double-logarithmic plots, the relative positions and slopes of which can be correlated with the variations in surface characteristics.
A. E. S.

Degradation of Cellulose by Ultrasonic Waves. K. Edelmann. *Faserforsch. und Textiltech.*, **4**, 407–412 (Oct. 1953).

A 0·5% gel in acetone of a highly polydisperse cellulose nitrate is irradiated with intense ultrasonic waves, frequency 800 kilocycles per sec.; the preparation is then fractionated and the D.P.-distribution curve determined. As irradiation proceeds, the proportion of high-D.P. (degree of polymerisation) fractions falls—thus the original polymer (average D.P. 1060) contains an appreciable amount of fractions of D.P. > 2500, but after irradiation for 4 hr. the average D.P. falls to 630 and a negligible proportion of the polymer is of D.P. > 1400. After this treatment the D.P.-distribution curve shows a peak at D.P. $= 1000$, and this maximum maintains its position and increases progressively in height as irradiation is prolonged further. It is considered that chain breakdown occurs until a limiting chain length is attained, molecules of this size being able to remain intact while following the vibrations of the solvent medium. Further experiments are carried out on dry cellulose in a vibratory mill, the cellulose being subsequently esterified with nitric acid and fractionated as before. The results show that in this case comminution proceeds indefinitely, there being no evidence of the attainment of a limiting chain length.
A. E. S.

Behaviour of Polycaprolactam in Hot Primary Aralkyl Alcohols and in Cresol. W. Dawydoff. *Faserforsch. und Textiltech.*, **4**, 412–417 (Oct. 1953).

It is shown that under the conditions necessary for the dissolution of polymerised ε-caprolactam in benzyl alcohol and in phenol–water and cresol–methanol mixtures, the polymer may generally be recovered (by pptn. with excess of methanol) without appreciable change in D.P., and some of the conditions, e.g. long standing in soln. at high temp., under which appreciable depolymerisation may occur are given. Solvent extraction of the low-D.P. components of the polymer preparation is discussed.
A. E. S.

Moths and Non-wool Textiles. S. Wolf. *Faserforsch. und Textiltech.*, **4**, 417–439 (Oct. 1953).

The habits of clothes moths (*Tinea pellionella* and *bisselliella*) are described in detail, and it is shown that their larvæ are able to attack indigestible fibrous materials of all kinds—including rayons, synthetic fibres, and even glass fibres—and may cause considerable damage in non-wool textiles under suitable conditions, e.g. when keratinous material is to be found in the vicinity. Numerous photomicrographs are given showing the comminuted fibres, as found in the excreta and as incorporated in larva cases and pupal cocoons. There are 75 references.
A. E. S.

Preparation and Properties of Cross-linked Polyacrylonitriles. A. Hunyar and H. Reinhardt. *Faserforsch. und Textiltech.*, **4**, 454–460 (Nov. 1953).

A number of cross-linked polymers are prepared by emulsion copolymerisation of *p*-divinylbenzene (1 mole) with acrylonitrile (1–580 moles), and the solubility and swelling properties (in dimethylformamide) are determined. The solubility falls off rapidly as the proportion of cross-linking component is increased, and none of the products is suitable for spinning into fibres. Similar results are obtained with other cross-linking agents (hexa-1:5-diene-3:4-diol and octa-2:6-diene-4:5-diol). No evidence of cross linking is found when acrylonitrile is polymerised in presence of peroxy compounds; the gel particles that form in practice and lead to difficulties in spinning are therefore considered to arise from other causes. In further experiments it is shown that cross linking readily occurs at high temperatures when a copolymer of acrylonitrile and acrylic acid (9:1) is treated in dimethylformamide soln. with a suitable agent (*p*-phenylenediamine, benzidine, quinol, urea), and on the basis of these results insoluble, cross-linked fibres are prepared by spinning a soln. containing cross-linking agent (which does not react in the cold), and then aftertreating the fibre at high temp. Cross linkage is brought about also by treating polyacrylonitrile fibres with a soln. of H·CHO in aq. H_2SO_4. Although in the latter case the elastic properties of fibres are not greatly affected, all the cross-linked fibres are lower in strength than normal polyacrylonitrile fibres.
A. E. S.

Concentration–Viscosity Curves as an Index of the Cross-linking of Perlon. A. Matthes. *Faserforsch. und Textiltech.*, **4**, 510–511 (Dec. 1953).

Sauer's work (see J.S.D.C., **69**, 220 (June 1953)) is criticised. It is considered that it affords no index of cross-linking in Perlon, though it may be true that oxidation leads to the formation of insoluble particles in the polymer melt. Sauer's z is not independent of the intrinsic viscosity $[\eta]$, in fact the author has shown (*J. prakt. Chem.*, **162**, 250 (1943)) that—

$$\eta = [\eta](c + 0{\cdot}25(\eta)c^2)$$

so that $z = 0{\cdot}25[\eta]^2$ and Sauer's x is identical with $[\eta]$. An examination of this equation shows that the value of z cannot be related to the alleged cross-linking, although it may be possible to explain any variation of the value of z/x from 0·25 by this means. This ratio does not, however, vary appreciably from this value over the range of concn. for which the equation is valid (up to 1% and not 5%, as assumed by Sauer).
A. E. S.

Fractionation of Synthetic Polymers for Fibre Manufacture. A. Gordijenko. *Faserforsch. und Textiltech.*, **4**, 499–507 (Dec. 1953).

It is considered that in the development and control of synthetic fibre production it is desirable to determine the D.P.-distribution curve for the polymer, and not merely the average D.P. (degree of polymerisation), as given by direct viscosity measurements. However, the D.P.-distribution curve alone does not always give adequate characterisation of a polymer preparation, for examination of certain properties—the time for a standard degree of turbidity to appear on addition of non-solvent, and the change in viscosity on standing—of solutions of Perlon or Perlon fractions, apparently identical in D.P. and method of preparation, reveals a great measure of variation. Methods of fractionating polymers are reviewed, and results obtained on polyamide fibres, Orlon and Terylene are examined critically. Fractionation methods developed by the author are described, and the methods of represent-

ing the results graphically are discussed. Fractionation of Perlon is effected by shaking a 4–6% soln. in cresol with sufficient light petroleum (aliphatic, olefin-free, b.p. 80–100°C.) to give a turbid liquid that separates into two clear layers within 30 min., all operations being carried out at 50°C. The upper layer contains a fraction of low D.P. (~5% of the Perlon taken) the separation of which is described; its D.P. is determined viscometrically. The lower layer is diluted with an amount of cresol equal to that removed in the upper layer, and the fractionation process is repeated. The procedure is repeated with slight variations until a full range (10–12) of fractions is obtained (one operator can do two such fractionations in 7–8 days). Similar partition methods are found to be satisfactory also for the fractionation of Orlon (in dimethylformamide at 50–60°C.) and Terylene (in phenol–tetrachloroethane at 65°C., but not in *m*-cresol, which gives non-reproducible results attributed to depolymerisation). Also, an attempt is made to fractionate Orlon by passing a benzene-dimethylformamide soln. of the polymer through an alumina column and fractionally eluting the adsorbate, but it is found that some depolymerisation occurs during this treatment. Work on improved fraction-precipitation methods for Perlon is reported. A. E. S.

Comparison between the Degradation of Cotton and Wood Pulp. O. Samuelson, G. Grangard, K. Jönsson, and K. Schram. *Svensk Papperstidning*, **56**, 779–784 (31 Oct. 1953).

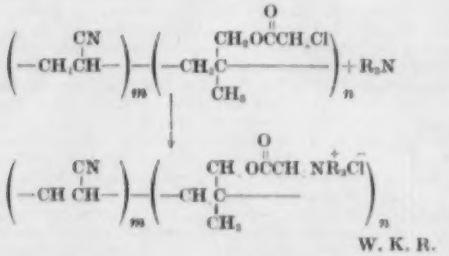
Cotton and wood pulp were degraded by means of acid hydrolysis, alkaline cooking at high temperatures, and ozone, and yield, viscosity, cold alkali solubility, hot alkali solubility and carboxyl no. determined. Differences in the sub-microscopic structure of cotton and wood pulp are explained on the basis of these investigations.

S. V. S.

Preparation of Dyeable Fibre-forming Compositions. Reaction of Acrylonitrile–Vinyl Chloroacetate Copolymers with Amines. G. E. Ham and P. W. Gann. *Ind. Eng. Chem.*, **45**, 2320–2323 (Oct. 1953).

Reaction of Acrylonitrile–Alkenyl Chloroacetate Copolymers with Amines. G. E. Ham, A. B. Craig and P. W. Gann. *Ind. Eng. Chem.*, **45**, 2323–2326 (Oct. 1953).

Copolymers of acrylonitrile (I), >85%, with vinyl chloroacetate (II) or methallyl chloroacetate (III) were prepared by suspension polymerisation with potassium persulphate as catalyst, and subsequently treated in dimethylformamide solution, with the stoichiometric quantity of trimethylamine for replacement of the active chlorine in the copolymers, thereby introducing basic groups along the polymer chain. The product was readily spun and stretched into fibres with good physical properties. Fibres thus prepared from a copolymer containing > 3% II or III could be dyed satisfactorily with acid dyes. Copolymers I–II were not entirely satisfactory because of partial hydrolysis of II during suspension polymerisation, and because of the tendency of the amine treated solution to thicken or gel. and I–III copolymers were preferred. Fibres of both types suffered appreciable discoloration on heating at 300°F. Replacement of III by allyl chloroacetate yielded a gel, after amine treatment, which could not be spun. Alternatively, the initial copolymers were spun and basified by treatment in fibre form with various amines or ammonia, but this procedure was not as effective as treatment in solution prior to spinning. It is thought that the reaction is—



W. K. R.

Polyethylene Terephthalate Films— Physical Properties. L. E. Amborski and D. W. Flierl. *Ind. Eng. Chem.*, **45**, 2290–2295 (Oct. 1953).

Polyethylene terephthalate is available in both fibre and film forms, the latter being known as "Mylar", and produced in thicknesses 0·00025–0·00075 in. The film, m.p. 255°C., d. 1·38, absorbs < 0·5% water on immersion and its physical properties are substantially the same wet and dry. It is tough and durable over a wide temp. range (−60°–200°C.), insoluble in most common organic solvents, but soluble in some polar solvents, e.g. phenol; resistant to mineral acids, but somewhat susceptible to strong alkalis. The film has a high dielectric strength and high resistivity, even at elevated temperatures and is relatively impermeable to water and solvent vapours, oils, greases and odours.

W. K. R.

Fibres. C. S. Grove, J. L. Vodonik, and R. S. Casey. *Ind. Eng. Chem.*, **45**, 2199–2304 (Oct. 1953).

A survey of recent developments with particular reference to the output and utilisation of synthetic fibres. An annotated bibliography (163 references) covering 1952–3, is appended. W. K. R.

Synthetic Fibres— Their Achievements and Potentialities. R. W. Moncrieff. *Rubber and Plastics Age*, **35**, 24–26 (Jan. 1954).

A review of the properties, uses, and potentialities of nylon, terylene, and many of the acrylic type fibres; their mechanical properties, and resistance to chemical and bacterial attack are virtually new to the fibre field. The low moisture absorption of these fibres gives good wet strength and ease of drying, but leads also to dyeing difficulties. In contrast to wool, which may absorb 30–40% of moisture without feeling damp, the synthetic fibres hold 1% in some cases, and nylon has the highest absorption (3·5%). A higher level of absorptivity is desirable in fibres which are, in the form of clothing, to remain in contact with the human body. Vinylon (or Kuralon), produced in Japan, is made by saponifying polyvinyl acetate and then hardening it by reaction with HCHO; it thus contains a number of OH groups and has a moisture regain of 5%. Developments along these lines are regarded as desirable. J. W. D.

PATENT

Acrylonitrile Polymers having Improved Dyeing Properties. Industrial Rayon Corp. *USP* 2,631,995

Copolymers of acrylonitrile and mono-alkyl or mono-methylalkyl ethers of saturated amino alcohols are readily dyed with acid dyes. C. O. C.

Solvents for Acrylonitrile Polymers (III p. 158).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Study of the Viscosity of the System: Water–Sodium Stearate–Alcohols in presence of Sodium Chloride. A. N. Bose. *J. Indian Chem. Soc.*, **30**, 496–498 (July 1953).

Data are presented which show the effect of temp. and concn. on the viscosity of the system water–sodium stearate–alcohols (*n*-butyl and *n*-propyl), to afford comparison with similar data published by the same authors (*Ibid.*, **29**, 43, 135 (1952)) relating to the soap of an unsaturated acid (sodium oleate). The behaviour of the two systems (oleate and stearate) is closely similar. With other alcohols white gels are formed at room temp., these becoming sols on heating, the transition temp. varying with the alcohol; these temp. were too high to permit comparative study within the scope of the work reported. In the case of *n*-butyl and *n*-propyl alcohols gels were formed at 5°C., these returning to sols at ca. 15°C. Such gels are not formed in sodium oleate systems. It is suggested that sodium stearate sols result from the combination of micelles to form larger micelles and therefore gels; the temp. of gel-formation depends upon the strength of the binding forces between the smaller sols for the alcohol present. J. W. D.

Laundering of Cotton. W. Fong and H. P. Lundgren. *Ind. Eng. Chem.*, **45**, 13A–15A (Oct. 1953).

It is reported that certain proteins and neutral synthetic polymers act in a similar manner to carboxymethylcellulose (CMC) in inhibiting the redeposition of soil during the laundering of cotton. Their effectiveness was assessed by the photometric measurement of the deposition of carbon black from detergent-alkali builder systems. Of the proteins, gliadin was highly effective, being rather better than CMC; a correlation was found between whiteness retention activity and proline content of the protein. The effectiveness of uncharged synthetic polymers depended on their molecular size; after an optimum size, soil deposition increased with increasing mol. wt. Partially acetylated low mol. wt. polyvinyl alcohol (PVA) and low mol. wt. polyvinylpyrrolidone (PVP) gave excellent results. Charged polymers, e.g. Na polyacrylate, were unsatisfactory. In all cases, whiteness retention was impaired by the presence of electrolytes— PVA being extremely sensitive, PVP relatively insensitive, with gliadin and CMC intermediate— and by lowering the pH.

W. K. R.

Detergency and Detergent Testing. R. E. Wolfrom and A. C. Nuessle. *Amer. Dyestuff Rep.*, **42**, P753–P762 (9 Nov. 1953).

Because of the large number of detergents on the market and the time-consuming nature of bulk trials, selection of the correct detergent for a particular job is difficult, and a discussion is given on factors to be considered in selecting a test method. Several of these are described, emphasis being laid on speed and ease of operation, and specimen results, including costing, are presented. Industrial processes studied are raw wool scouring, and kier boiling. Apart from problems connected with natural grease removal, experiments on removal of artificial soiling are also described, including effects of soil type, soil loading, and form. An interesting quantitative study is made of the effect of energy input on soil removal, using a machine in which a vertical-motion washing head contains a disc of soiled cloth and is driven over a range of 0–200 cycles/min.

J. W. B.

VIII— DYEING

Wool Dyeing at Elevated Temperatures. J. J. Iannarone, H. F. Clapham, and R. J. Thomas. *Amer. Dyestuff Rep.*, **42**, P666–P671 (28 Sept. 1953).

The blending of wool with fibres such as Orlon and Terylene, which are advantageously dyed at high temperatures, lends interest to the dyeing properties of wool under similar conditions. Accordingly, small amounts are dyed in glass "bombs", immersed in a polyethyleneglycol bath, and larger quantities in 1 lb. Smith-Drum and 2 lb. Venago machines adapted to a pressure system. Hand-knitting yarn is treated at different temperatures and for different times with mixtures of Na_2SO_4 and H_2SO_4, HCOOH, and CH_3COOH, followed by strength measurements on the Instron tester. Treatment at 250°F. is hazardous, particularly with H_2SO_4, even for 10 min.; 225°F. appears safe, though neutral baths lead to loss of elongation. Carpet yarns give similar results, in fact 225°F. values are better than those obtained after normal dyeing, which may be due to the shorter treatment which suffices at the higher temperature. Strengths of shades obtained at 225°F. are generally equal to or better than those at the boil. Exhaustion still requires acid if it is to be complete. Levelling may or may not be improved, according to the dye selected; some milling dyes are unchanged but most chrome dyes show improvement. It is suggested that the greatest advantages in practice are to be obtained with mill routines which involve long dyeing cycles, e.g. chrome dyeing of tops; figures are quoted showing great savings in time. Photomicrographs of cross-sections taken from mill-scale dyeings indicate greatly improved penetration.

J. W. B.

Dyeing Orlon Acrylic Fibre and Terylene Polyester Fibre, and Blends with Other Fibres. J. F. Laucius. *Amer. Dyestuff Rep.*, **42**, P792–P796 (23 Nov. 1953).

A survey is presented of recent advances in dyeing Orlon and Terylene, with special emphasis on their blends with other fibres. Particular attention is given to basic dyes on Orlon, and the cuprous ion method is reviewed especially with regard to reducing agents, selection of dyes, and dyeing temperature. Blends include Orlon–wool, and Orlon–rayon or cotton; and Terylene–wool, and Terylene–cellulosic.

J. W. B.

Problems of Perlon Dyeing. E. Heilmann. *Faserforsch. und Textiltech.*, **4**, 493–499 (Dec. 1953).

General review of Perlon dyeing— available dyes, dyeing behaviour, effect on dyeing of various processes, e.g. setting, etc.

A. E. S.

Theory of Ionic Absorption on Wool Keratin. B. Olofsson. *J. Polymer Science*, **12**, 301–308 (Jan. 1954).

The Donnan theory does not give a correct explanation of ionic absorption in wool keratin, because the anions are not randomly distributed in the "internal phase" as "gegenions" of the fixed charges. They are really adsorbed, e.g. fixed to localised electrovalent sites. For Cl^- and SO_4^{2-} the adsorption energy is of about the same magnitude as the adsorption energy of water. These results are probably not confined to wool but should be considered for high polymers where there is a possibility to get "localised sites" for positive as well as negative ions. The original Donnan theory is only valid for (*a*) membrane equilibria, (*b*) absorption of "gegenions" having no "localised sites". It should be possible to determine the type of fixation (number of sites per molecule) of anions of dyes or detergents by comparing their experimental absorption in mixtures with calculated absorption from molecular statistical considerations.

C. O. C.

The Artificial Colouring of Foodstuffs. D. W. Grover. *Food Preservation Quart.*, **13**, 32–37 (1953); *Chem. Abs.*, **48**, 295 (10 Jan. 1954). A review.

C. O. C.

Leather Dyes— a Survey (XII p. 170).

IX—PRINTING

Photographic Colour Development. I— Rate of Colour Development. Y. Tomada and M. Kawasaki. *Repts. Govt. Chem. Ind. Research Inst., Tokyo*, **47**, 177–186 (1952); **II— Reducing Properties of *p*-Aminodiethylaniline.** *Ibid.*, **48**, 1–11 (1953); *Chem. Abs.*, **48**, 63 (10 Jan. 1954).

I— Positive films were developed at 20°C. for 2-32 min. in a Dewar flask with a developer of pH 8·24 made of *p*-$NH_2C_6H_4N(C_2H_5)_2{\cdot}H_2SO_4$, Na_2SO_3, and borax. The following colour couplers were used: 1-naphthol, dibromo-1-naphthol, thymol, 1-*p*-nitrophenyl-3-methyl-5-pyrazolone, *p*-$NO_2C_6H_4CH_2CN$, $CH_3CO{\cdot}CH_2CONHC_6H_5$, $CH_2{\cdot}(COOC_2H_5)_2$ and $C_6H_5COCH_2COOC_2H_5$. The developing velocity constant ($k = 1/t$, log $\gamma_1(\gamma_2-\gamma_1)$ calc. for $t_1 =$ 16 min.; γ_1 and γ_2 = slopes of developing (blackening) curves for 16 and 32 min. respectively) was increased slightly by addition of couplers, except for 1-naphthol which fogged the films, and the pyrazolone which decreased k. The developer after being brought to pH 10·2 with Na_2CO_3 showed higher but more varied (with different couplers) From the temperature coefficient of k determined at 10–30°C. for both developers without a coupler the apparent activation energy was calculated to be 9·60 K. cal./mole.

II—The relative reducing value (= mol. AgBr reduced per mol. reagent) of *p*-$NH_2C_6H_4(C_2H_5)_2$, H_2SO_4 and the corresponding hydrochloride was about 3. In $AgNO_3$ soln. the sulphate reduced $AgNO_3$ in the ratio 1 : 2 mol. at the maximum; the hydrochloride 1 : 4 mol. (at the same pH). The oxidation–reduction potential of the sulphate (determined in various buffers made of NaOH, H_2BO_3 and KCl) dropped linearly with higher pH and with the log of concentration. The potential changed similarly in the sulphate after adding various amounts of Na_2CO_3, and dropped rapidly after adding Na_2SO_3 especially at its higher concentrations.

C. O. C.

PATENT

Aqueous Emulsions of Pigmented Lacquers for Colouring Textiles. Sun Chemical Corpn. *USP* 2,631,985

Oil-in-water emulsions which can be diluted either by water alone or by water, oil and uncoloured emulsions to

bring to the strength desired for use contain ≮ 15% of binder and the ratio of binder to pigment must not be less than 15:12 nor more than 15:8·5 by weight. In addition the ratio of binder to the plasticiser-emulsifier mixture must not be less than 15:9·5 nor greater than 15:7·5 by weight. C. O. C.

X—SIZING AND FINISHING

Finishes for Cellulosic Textiles giving Permanent Protection against Microbiological Attack—A Survey of the Literature. M. Hamlin. *J. Textile Inst.*, **44**, P 745–P 753 (Nov. 1953).

A review of the literature on antibacterial finishes for cellulosic fibres, with 44 references. J. W. B.

Nitrile Rubber Latex as Permanent Abrasion-resistant Finishes for Blue Denim Fabric. E. Abrams and N. Sherwood. *Amer. Dyestuff Rep.*, **42**, 777–782 (23 Nov. 1953).

Blue denim fabric is selected for abrasion-improvement as it is normally made from short-staple cotton and is dyed with indigo, which remains on the surface and is easily removed by abrasion. After preliminary trials indicated that improvement is obtained by sizing the fabric with a latex of acrylonitrile-butadiene mixture, the process is applied to warps as a substitute for the normal starch sizing. The abrasion resistance of the final fabric increased with laundering, being after 5 launderings twice as great as originally. Under similar conditions starch-sized fabrics decrease in resistance until, after 5 launderings, their resistance is that of desized fabrics. The latex finish also prevents colour bleaching during laundering, and crocking and colour transfer during laundering and ironing. White fabrics remain unimpaired when so processed in contact with latex-sized material. Microscopic examination shows that the latex surrounds the yarn in a net-like structure, and is perhaps progressively cured with each ironing, thus improving the abrasion resistance. J. W. B.

PATENTS

Permanent Waving. E. O. Schnell. *USP* 2,631,965

An aqueous alkaline solution containing alkali or ammonium thioglycollates together with a water-soluble, non-reducing ammonium salt, e.g. ammonium chloride, sulphate, bisulphate or formate, is used as the sole processing solution. The solution is so adjusted that at the start it is at pH 9·2–9·5 and then after application the pH falls steadily to pH 4·5 when the waving is completed. C. O. C.

Bonded Open-mesh Web. International Cellucotton Products Co. *BP* 703,345

Open-mesh web suitable for covering disposable towels etc. is produced by coating a fine, e.g. 18 × 8, open-mesh cloth with adhesive in small discrete areas at intersections of the threads and also discontinuously along the threads. A facing of non-woven fibre, e.g. cotton, is then applied and anchored to this face, thereby greatly strengthening the original mesh, improving its appearance, and retaining fluid permeability. J. W. B.

Chenille Pile Fabrics—Manufacture by Bonding to Weft Threads. G-C, Ahier, A. V. Champagnat, and J. F. M. Tirmont. *BP* 702,460

A warp of chain threads is treated on one side with a bonding agent and arranged in tubular formation; the tube is supported on the inside and weft threads are wound round the outside at a speed suitably adjusted to the rate of chain feed. Bonding is then effected by heating and the tube is slit to form a flat mesh. By cutting this between each warp chain thread, and passing them through heated V grooves a U-shaped chenille thread is formed. This can subsequently be applied to a mesh backing to form carpets or other pile fabrics, using a thermosetting bonding agent. J. W. B.

Coating Textiles, Paper, etc. with a Continuous, Impervious Film of Organic Material. American Viscose Corpn. *USP* 2,631,958

When transferring a continuous film of organic material from a backing to fabric, etc. by means of heat and pressure breaking of the film is avoided by interposing between the fabric and the film a layer of thermoplastic material which fills the interstices in the fabric, lays the nap and partly coats the fabric surface. Preferably the interposing layer becomes tacky at a lower temperature than does the coating film. C. O. C.

Cotton—An Old Fibre with a New Future (VI p. 164).
Moths and Non-wool Textiles (VI p. 165).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Aromatic Part of Native Lignin. K. Kürschner. *Faserforsch. und Textiltech.*, **4**, 461–464 (Nov. 1953).

Recent work (F. Schutz *et al.*, *Cellulose-Chemie*, **21**, 55 (1943); **22**, 1, 114 (1944) *Z. angew. Chem.*, **60**, 63, 115 (1948); F. Reiff, *Z. angew. Chem.*, **60**, No. 3 (1948)) has indicated that native lignin is predominantly carbohydrate in character, appreciable aromatic character making its appearance only in the course of isolation. It is considered that, if Schütz's view of native lignin as a methylated anhydro-polysaccharide is correct, then, since reaction with water will be necessary before the lignin can be depolymerised to monomer as the first stage in its conversion to aromatic isolable lignin, nitration in alcohol in absence of water of completely water-free wood could not possibly yield an aromatic cpd. similar to "nitrolignin". Actually, nitration with careful exclusion of water is found to lead to the formation of some nitrolignin, and it is concluded that native lignin, though predominantly a polysaccharide derivative, is partly aromatic (1 part in 3 for the lignin of spruce; 1 in 7 for that of beech). A. E. S.

Reduction of Oxycelluloses by Sodium Borohydride. A. Mellor. *Chem. and Ind.*, 1204 (7 Nov. 1953).

The resistance of "gluconic acid oxycellulose" (I) to fragmentation and dissolution by hot 1% alk. soln. is decreased by reduction with Na borohydride, and restored by subsequent reoxidation by chlorous acid. The dominant influence in this connection of –CO groups present in oxycellulose is demonstrated. Results also indicate that (I) may contain lactonised –COOH groups which are readily reduced to –CO, whereas dichromate oxycellulose contains free –COOH groups which are not readily reducible by Na borohydride. J. W. D.

Degradation of Dextran in an Alternating Electrical Field. F. G. Fautard. *Chem. and Ind.*, 1316–1317 (5 Dec. 1953).

Macromolecular dextran is degraded when, at pH 7, its aq. soln. is subjected to an alternating potential between two electrodes immersed in it. Cooling becomes necessary as soln. temp. rises, and there is a steady fall in viscosity until a steady state (analogous to the observed end-point of ultrasonic degradation) is reached, after which the decrease in viscosity is very gradual. No change in pH is observed. Unless reducing agents are added, some fragments of the degradation are oxidised and rendered insol. Degradation is not confined to the electrode faces although some electrodes such as stainless steel are rapidly eroded (carbon is unaffected). Distribution of the degraded fragments, which is influenced to some extent by the shape and position of the electrodes, is shown to resemble closely that of an ultrasonically degraded dextran of equal mean viscosity, and both are markedly different in this respect from an acid-hydrolysed dextran. The mechanism of the breakdown is little understood. As with ultrasonic degradation, the initial rupture of the large mol. is a thermal process, the chemistry of the final fragments being largely determined by the proximity and character of adjacent ions. J. W. D.

Influence of some Pulp Properties upon Dissolution of Cellulose Xanthate. O. Samuelson and H. Jonsén. *Svensk Papperstidning*, **56**, 866–873 (30 Nov. 1953).

The filterability of viscoses from various chemical pulps was compared with the dissolving of the pulp in emulsion xanthation. With sulphite pulp the filterability deteriorated with increasing viscosity, whilst sulphate pulp clogged the filter to a greater extent than sulphite pulp. By increasing the addition of CS_2 to the extreme, a particularly good filterability could be obtained with

sulphate pulp. There was a relationship between the filtering properties and the dissolving in the course of emulsion xanthation, but with mercerised pulp the optimum filterability was obtained at a lower concentration of steeping liquor than with ordinary pulps. In emulsion xanthation a higher concentration of liquid is needed to bring the mercerised pulp into solution. S. V. S.

Combined Calcium in Rayon Pulp and its Behaviour in the Viscose Process. III—Precipitation from the Alkalising Liquor. T. N. Kleinert and W. Wincor. *Svensk Papperstidning*, **56**, 874–875, (30 Nov. 1953).

In the alkaline treatment of rayon pulp the calcium existing in the form of the carbonate of the hemicellulose complex reacts with the sodium carbonate portion of the liquor to yield calcium carbonate which separates out in a finely divided form and is adsorbed on the fibres and the precipitated hemicellulose colloids, bringing about clogging of the filter when the alkali cellulose is pressed. This happens particularly if part of the alkalising liquor is reused without previous careful filtration. In contrast to the amount of calcium introduced into the system with the pulp and the fresh liquor, the combined calcium of the pulp is of prime importance in causing the trouble described. S. V. S.

Effect of a Disintegration during Sulphite Cooking. O. Samuelson and A. Wennerblom. *Svensk Papperstidning*, **56**, 745–750 (15 Oct. 1953).

Chips were cooked until soft enough to disintegrate easily, defibred in a disintegrator, and cooking continued. The dissolution of lignin and wood polyoses, and the opening up of the cellulose was quicker than with normal sulphite cooking and the yield and properties of the pulp did not suffer if a short second cook was used. The pulp dissolved more easily in emulsion xanthation than ordinary sulphite pulp. S. V. S.

Carboxymethyl Cellulose in Free Acid Form. S. F. Dieckman, J. G. Jarrell, and R. S. Voris. *Ind. Eng. Chem.*, **45**, 2287–2290 (Oct. 1953).

Aqueous solutions of the sodium salt of carboxymethyl cellulose (CMC) may be converted to stable dispersions of CMC free acid, by treatment with a strong acid type cation exchange resin, e.g. Amberlite IR–120. (sulphonated styrene-divinylbenzene copolymer). On drying, the dispersions give films that are insoluble in water, but soluble in alkalis, pH > 11. The films may be plasticised with glycerol. The properties of CMC free acid suggest its possible use as an adhesive, permanent textile size, scaffolding fibre, and pigment binder. W. K. R.

Sulphite Pulp from Beech Bark and its Behaviour in the Viscose Process. T. N. Kleinert and P. Wurm. *Svensk Papperstidning*, **57**, 19–22 (15 Jan. 1954).

Beech bark was subjected to a normal sulphite cook and the composition of the product determined. The cooked bark, treated by the usual viscose process, yielded a viscose of about 5% NaOH content. It was shown that the cellulose fibres of the bark bast are completely dissolved in the viscose process but not the highly mineralised stone cells. These resist not only the sulphite process but also the viscose process, and can be detected in the residues of technical viscose filtration. Bark residues from imperfectly or badly barked wood can therefore give rise to considerable trouble in the viscose process, particularly in filtering and spinning. S. V. S.

PATENTS

Applying a Water-Insoluble Coating or Film to Paper or the like. Svenska Cellulosa. *BP* 703,717

The fibrous material, e.g. paper, is treated first with an alkali solution of a water-insoluble alkali metal salt of carboxymethylcellulose and then with an agent which causes decrease in pH and so precipitates the carboxymethylcellulose salt on the fibrous material. S. V. S.

Impregnation of Fibrous Materials such as Cardboard, Wallboard and Chipboard. Aktiebolaget Statens Skogsindustrier. *BP* 704,097

The fibrous web is treated on one side with an impregnating agent and hot pressed against a polished plate, or between a polished plate and a wire mesh, the treated side being made acid in reaction at least prior to the completion of the pressing, thereby causing the impregnating agent to flow wholly or partially through the thickness of the web, according to the quantity and viscosity of the agent, without adhering to the polished plate, and at the same time forcing water present in the web through the other side of the web, finally hardening at 150–200°C. S. V. S.

Impregnated Pressed Fibrous Materials having a Coloured Surface. Aktiebolaget Statens Skogsindustrier. *BP* 704,101

The impregnating agent is applied to the fibrous material when the latter is in the form of a continuous fibre web but before it has received its final form by pressing under high pressure against a polished plate, or between a polished plate and a wire mesh, as claimed in *BP* 704,097 (above) in which either simultaneously with the application of the impregnating agent, or subsequently in connection therewith, a colouring agent, e.g. a pigment, is applied to the side of the fibre web turned towards the polished plate. An inorganic compound, e.g. silicic acid, a silicate, or aluminium hydroxide, may be added which possesses such surface-active properties that it can serve as an interface active substance between the impregnating agent and water, with water as the continuous phase. S. V. S.

Regenerated Cellulose Film. British Cellophane. *BP* 703,416

A process for manufacturing regenerated cellulose film, treated on one side only with colloidal resinous material, comprises bringing two purified regenerated cellulose films in the gel state into continuous intimate contact and treating them with an aqueous dispersion at pH 6–7·5 containing hydrophilic colloidal particles of a precondensate of e.g. urea–formaldehyde, thiourea–formaldehyde, etc., drying the assembly without curing the resin and finally separating the two films.

Laminated sheet material comprises the film secured to a cellulosic or metallic sheet with an acidified thermoplastic adhesive applied to the treated side of the regenerated cellulose film. The film may be printed on the treated side by applying a printing ink containing an acidic substance and heating to cure the resin. S. V. S.

Degradation of Cellulose by Ultrasonic Waves (VI p. 165).
Comparison between the Degradation of Cotton and Wood Pulp (VI p. 166).
Coating Textiles, Paper, etc. with a Continuous Impervious Film of Organic Material (X p. 168).
Aminoplastics—Recent Trends, with Special Reference to their Application to Paper (XII p. 170).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Kinetics of Polypeptide Formation from *N*-Carboxy-α-amino Acid Anhydrides. D. G. Ballard and C. H. Bamford. *Nature*, **172**, 907–908 (14 Nov. 1953).

It is found that, when working with a constant pressure apparatus, the polymerisation of sarcosine *N*-carbonic anhydride in nitrobenzene solution is strictly a first order reaction, but is strongly catalysed by carbon dioxide. This explains earlier results giving a different order of reaction, and a reaction mechanism is suggested. Polymerisations of *N*-carbonic anhydrides of DL-phenylalanine and DL-leucine in nitrobenzene solution are completely different from that of sarcosine, and CO_2 has no catalytic effect. J. W. B.

Interaction of Vegetable Tannins with Polyamides as Proof of the Dominant Function of the Peptide Bond of Collagen for its Binding of Tannins. K. H. Gustavson. *J. Polymer Science*, **12**, 317–324 (Jan. 1954).

Polyamides have marked affinity for vegetable tannins and bind them irreversibly, showing that the peptide linkages function as binding sites for vegetable tannins. This fixation is independent of the pH of the system in the pH range 2–8. Identical independence is shown in fixation of tannins by collagen from solutions containing NaCl, which prevents swelling. The typical fixation curve of vegetable tannins by intact collagen is very dependent on pH: in this case the tannin fixation is directly related to the degree of swelling of collagen as a function of the pH

of the system. This is because the degree of swelling of the protein governs the number of coordination-active peptide bonds present in collagen for fixing tannins. Esterified collagen, in which most of the potential hydrogen-bonding sites are free to react, fixes large amounts of tannins independently of the hydrogen ion concentration in the range pH 2–7. The rapid drop in fixation of tannins at pH > 8 is caused by ionisation of phenolic groups, leaving fewer hydrogen bonds available on the tannin molecule. It seems that the main reaction of polyphenolic tannin of the mimosa type consists in condensation of a polyphenolic molecule for peptide bonds of adjacent protein chains, probably by means of hydrogen bonds. This multipoint attachment explains the stabilisation of the protein lattice effected by the tanning agent.

C. O. C.

Soaking Leathers—Round Table Discussion. *J. Amer. Leather Chem. Assocn.* Supplement No. 2 (4 June 1952).

Uni- and Multi-point Binding of Chromium Complexes by Collagen and the Problem of Cross-linking. K. H. Gustavson. *J. Amer. Leather Chem. Assocn.*, **48**, 559–579 (Sept. 1953).

Experimental evidence of cross-linking in globular proteins and of cross-linking collagen by tanning agents is surveyed and discussed, and a study is made of the interaction of cationic basic chromium salts with gelatin in dilute solution. From solutions of basic chromium chlorides the cation $(Cr_2(OH)_2)Cl_2^+$ is unifunctionally fixed by gelatin; of the corresponding basic sulphate 80% is fixed as unifunctional cations of the type—

$$\left(\diagup Cr_2(OH)_2SO_4\right)_2^{SO_4^+}$$

and the remainder as

$$\left(\begin{matrix}\diagup \\ Cr_2(OH)_2(SO_4) \\ \diagdown\end{matrix}\right)$$

complexes by two protein –COOH groups. With regard to normal tanning, in hide powders tanned with the basic sulphate to 11% Cr_2O_3 content, on the basis of collagen, about 90% total Cr is introduced as unifunctional complexes and the rest, representing ~1% Cr_2O_3, is bifunctionally combined. Thus only a very small fraction of the Cr fixed by collagen is effecting stabilisation of the structure by means of cross-linking. Initial chrome fixation preferentially involves carboxyl ions of adjacent chains, which sterically are able to combine with one Cr complex. The main part of the Cr complexes in later stages of tanning rupture and eliminate ionic cross links by unipoint attachment to salt-link carboxyl groups. J. W. B.

Organic Chemistry of Vegetable Tannins. A Survey of 50 Years. R. M. Lollar. *J. Amer. Leather Chem. Assocn.*, **48**, 656–673 (Nov. 1953).

Syntan Development in the U.S.A.—A Survey. T. Blackadder and I. C. Somerville. *J. Amer. Leather Chem. Assocn.*, **48**, 673–681 (Nov. 1953).

Leather Dyes—A Survey. C. K. Black and C. H. Geister. *J. Amer. Leather Chem. Assocn.*, **48**, 682–689 (Nov. 1953).

50 Years of Leather Colouring. R. H. Leach. *J. Amer. Leather Chem. Assocn.*, **48**, 605–612 (Oct. 1953).

A brief survey of leather dyeing over the past half-century. J. W. B.

Depilation of Sheepskins with Enzymes. J. M. Gillespie. *J. Soc. Leather Trades Chem.*, **37**, 344–353 (Nov. 1953).

Samples of sheepskin are depilated with culture filtrates of *A. parasiticus* in presence of activating chemicals. Effective depilation takes place over a fairly wide pH range, with maximum action at pH 7·0. Optimum temperature is about 35°C. but effective depilation is obtained below this, with longer incubation. Efficiency increases with concentration of enzyme; Na_2SO_3 is necessary but the amount required varies for different enzyme preparations and for different skins. High concentration may inhibit action either by inactivating the enzyme or by a salting-out effect in the substrate. Dialysis removes enzyme activity which is subsequently restored by adding reducing agents. No conclusive results are obtained in a study of divalent ion effect, and a list of inhibitory chemicals is included. With 2% enzyme protein in a mixture of 0·1 M. Na_2SO_3 and 0·5 M. $ZnCl_2$, thickened with clay, loosening is complete in 4 hr. at 30°C, and at pH 7.

Other moulds are studied and *A. fumigatus, A. effusus, A. ochraceus, A. wentii* and *P. griseo-fulvum* all show depilatory activity; *A. oryzae* is more active than *A. parasiticus* and does not require a reducing agent. Culture filtrates of bacteria isolated from sweated sheepskin are less effective than *A. parasiticus* and require a disulphide-bond splitting agent. Tissue extracts from pig pancreas, although not requiring an activator, are less active than the *Aspergilli*. J. W. B.

Origin of the Absorption Band at 1550 cm.$^{-1}$ in Proteins. H. Lenormant and E. R. Blout. *Nature*, **172**, 770–771 (24 Oct. 1953).

The protein adsorption band at 1550 cm.$^{-1}$ is probably a complex absorption band associated in part with $-COO^-$ groups (1575 cm.$^{-1}$) and in part with two kinds of peptide linkings. One type is easily deuterated at room temp. and the other at high temps. or in alkaline solution. J. W. B.

Sheepskin Degreasing—Comparison of White Spirit and Paraffin. R. G. Mitton and K. G. A. Pankhurst. *J. Soc. Leather Trades Chem.*, **37**, 331–339 (Oct. 1953).

Sheepskins for gloving leather are degreased in the pickled state with pool burning oil, vaporising oil, white spirit, and two experimental white spirits of high and low aromatic contents. There is no great difference with regard to degreasing efficiency and leather quality, but grease and solvent recovery is greatly facilitated by lower boiling-range solvents such as the white spirits. J. W. B.

Action of Large Ions on Collagen and Gelatin. K. G. A. Pankhurst. *J. Soc. Leather Trades Chem.*, **37** 312–320 (Oct. 1953).

The action of anionic and cationic detergents on collagen and gelatin is described and discussed, and the experimental evidence applied to leather making processes. J. W. B.

XIII—RUBBER; RESINS; PLASTICS

Aminoplastics—Recent Trends, with special Reference to their Application to Paper. F. L. Hudson and C. P. Vale. *Trans. Plastics Inst.*, **21**, 5–26 (Oct. 1953).

The development of stable aq. soln. of urea– and melamine–HCHO resins of low mol. wt. has made possible a variety of textile finishing processes. In the production of cellulosic fabrics of crease and shrinkage resistant finish, there is excellent penetration of the fibre, though care is essential during drying to ensure that the resin does not migrate to the fibre surface where it will impair the handle. More highly condensed resins are retained on the outside of the fibres and, either alone or in admixture with starch, may be used to give permanent stiffness to a fabric. Durable and wash-fast prints are yielded by emulsions containing amino resins and into which pigments and/or metal powders are ground; the prints are heat-cured. By co-condensing a methylated methylol melamine with a reagent such as methylol stearamide, cellulosic fabrics may be given creasing, shrinkage, and water-resistance in a single operation. Other effects obtainable include permanent glazing and embossing, fire-proof finishes (by inclusion of phosphates), and, by the localised application of the resin followed by shrinkage, crimped effects. Melamine resins have been successfully used to control the shrinkage of wool; the mechanism of the effect is not generally agreed. Cellulose film used for packaging may be treated with a very small amount of melamine resin (from an acid colloid soln.) or cationic urea resin; seals made by means of nitrocellulose—alkyd resin lacquer then remain intact under damp conditions. The use of amino resins in conferring wet strength upon paper is described at length. Other applications briefly discussed include mouldings, laminates, adhesives, and coatings. It is claimed that the stain resistance of melamine–HCHO surfaces is enhanced if a melamine: HCHO ratio of 1:1·5 is used; further improvement results from the inclusion of benzoguanamine–HCHO resin. There are 70 references to the literature. J. W. D.

Solvents for Acrylonitrile Polymers (III p. 158).

Behaviour of Polycaprolactam in Hot Primary Aralkyl Alcohols and in Cresol (VI p. 165).

Preparation and Properties of Cross-linked Polyacrylonitriles (VI p. 165).

Acrylonitrile Polymers having Improved Dyeing Properties (VI p. 166).

XIV—ANALYSIS; TESTING; APPARATUS

Surface Colorimeter as an Aid to Standardisation of Leather Finishes. J. S. Mudd and F. E. Downs. *J. Soc. Leather Trades Chem.*, **37**, 353–362 (Nov. 1953).

It is difficult to ensure that successive batches of colour finishes are the same as those previously produced, and attempted standardisation using trichromatic filters similar to those used in photographic colour separation proved unsuccessful. Experiments are described in which satisfactory results are obtained with a photoelectric surface colorimeter, using a controlled light source and 3 composite and carefully calculated filters, in conjunction with a barrier layer photocell. Galvanometer readings are converted with the aid of tables into the conventional x, y, and z terms of the C.I.E. system, and reproducible colour measurements are made with an accuracy corresponding to that with which colours may be visually discriminated under normal conditions. A graphical procedure is suggested wherein an area is plotted within which there is no perceptible difference in shade from a predetermined standard when viewed by the average observer; matchings will then have to come within this area. While not suggesting that works practice can in all instances attain the high accuracy possible by this method, a working compromise is suggested. Effects of exposure to light are also studied in the hope that results more quantitative and generally useful than currently practised visual fading assessment would be obtained, and preliminary results are described. A routine procedure is given for spraying white card with colour prior to photoelectric analysis and filing for reference. J. W. B.

Gums in Black Wattle Extract—Determination of D. G. Roux. *J. Soc. Leather Trades Chem.*, **37**, 374–377 (Nov. 1953).

A tentative method for the quantitative determination of gums in black wattle extract is described. The gums are precipitated with absolute alcohol and washed with 95% alcohol to remove sugars and tannins, all being done in a centrifuge tube, and are quantitatively transferred with hot water to a silver basin and dried as for non-tannins. The method applies to studies on the adsorption of gums by hides during tannage. It is found that 45% of the gums present in commercial mimosa extract are adsorbed by hide pieces during a countercurrent experimental tannage. J. W. B.

Ethylene Oxide Products in Aqueous Solution or Dispersion—Quantitative Determination. N. Schönfeldt. *Nature*, **172**, 820 (31 Oct. 1953).

To a certain volume of aqueous solution or dispersion of ethylene oxide product, HCl is added with stirring, followed by a known quantity (a) of potassium ferrocyanide. A flaky precipitate is formed and is filtered, and excess ferrocyanide in the filtrate (b) is estimated by titration with $ZnSO_4$. $(a-b)$ is the quantity of ferrocyanide consumed by the precipitate. For each ethylene oxide condensation product a fixed relation exists between the quantity precipitated and the amount of ferrocyanide required, and can be determined (f) by using a known quantity of ethylene oxide product. For a solution containing an unknown amount (x) it follows that $x = f(a-b)$. Determinations carried out on nonylphenol condensed with 6, 9, and 12 moles of ethylene oxide, in varying dilutions, give values of f constant within $\pm 5\%$, which would be adequate for practical purposes. A relationship is also found between the number of moles of ethylene oxide present in one mole of condensation product and the number of moles of $K_4Fe(CN)_6$ required for precipitation. J. W. B.

Potentiometric Studies in Oxidation-Reduction Reactions. XIV—Oxidation with Alkaline Potassium Permanganate. B. Singh and G. Singh. *J. Indian Chem. Soc.*, **30**, 488–490 (July 1953).

Alkaline $KMnO_4$ is used, according to the method of Stamm (*Z. angew. Chem.*, **47**, 191 (1934); **48**, 710 (1935)), in the potentiometric estimations of KI, KIO_3, KCN, CH_3OH, glycerol, phenol, and salicylic acid. Excess $KMnO_4$ is back-titrated potentiometrically against standard sodium formate. The reaction is discussed, the method detailed, and titration curves shown. J. W. D.

Comparative Sunlight and Fadeometer Tests of Selected Dyed Manufactured Fibre Samples. A.A.T.C.C. Committee Report. *Amer. Dyestuff Rep.*, **42**, P748–P752 (9 Nov. 1953).

75 synthetic fibre samples (cellulose acetate, Acrilan, Terylene, dynel, nylon, and Orlon) are dyed with a selection of dyes and exposed in two steps, under glass and in direct sunlight. Of these 22 are selected and exposed in four steps by 10 laboratories. Results are tabulated, and in an appendix are described the purpose, scope, and operation of the grey scale for evaluating changes in colour. J. W. B.

Large-scale Chromatographic Fractionation of Tannin. W. E. Hillis. *J. Soc. Leather Trades Chem.*, **37**, 399–404 (Dec. 1953).

A preliminary investigation shows that wattle extract may be fractionated on a large scale by means of adsorption chromatography, using Magnesol and the liquid chromatogram technique. J. W. B.

Black Wattle Tannins in Spent Tannery Liquors—Determination. D. G. Roux. *J. Soc. Leather Trades Chem.*, **37**, 404–407 (Dec. 1953).

Rapid and simple means of tannin determination, suitable for small tanneries with no laboratory facilities, are required, particularly for spent black wattle tannin liquors where barkometer measurements may be erroneous. A new method is described depending on an intense blue-violet colour formed by Mitchell's ferrous tartrate reagent with the *o*-hydroxyphenolic groups of tannins, in presence of a buffer. Nessler tubes are used for colour comparison. A spot test is also described, for the very approximate assessment of tannin contained in spent liquors. J. W. B.

Detergent Evaluation. R. E. Wagg. *J. Textile Inst.*, **44**, T 658 (Dec. 1953).

As the chopped fibre method for evaluating detergents measures the extent of the removal of the solid component of the soiling mixture only it cannot necessarily be inferred that the oily component is removed in the same proportion. The column-tray method of Dickinson and Palmer (*Ibid.*, **42**, T6 (Jan. 1951)) is therefore used as a rapid means of determining oil content of washed chopped fibres. It is thereby shown that a detergent which produces an excessive number of "yellow centres" in laundered articles removes much less oil than a more satisfactory detergent, and that increasing the pH value of a soap solution increases oil removal. The column-tray method thus provides a useful extension of the chopped fibre method for evaluating detergents. J. W. B.

Determination of the Fatty Matter in Wool by Ether Extraction. F. F. Elsworth and J. Barritt. *J. Textile Inst.*, **44**, P754–P759 (Nov. 1953).

The Technical Committee of the International Wool Textile Organisation has drafted a specification for determining fatty matter in tops by ether extraction, certain conditions being imposed with regard to the usual variables in such procedures. Experiments are therefore carried out to assess the importance of these variable factors in relation to a specification. Number and rate of syphonings, and in particular the solvent-material ratio in the extraction chamber are studied. As small samples gradually pack down in the barrel it is unnecessary to specify one sample weight but only a maximum weight for any one size of barrel—a minimum ratio of 10:1 is suggested. Removal of oil from dry combed top is slow and incomplete and the number of syphonings is therefore important; with oil-combed tops it does not matter and the proposed specification appears in this respect to be satisfactory. J. W. B.

Application of the Polarograph to the Paint Industry. W. P. Georgans. *J. Oil and Col. Chem. Assocn.*, **37**, 84–100 (Feb. 1954).

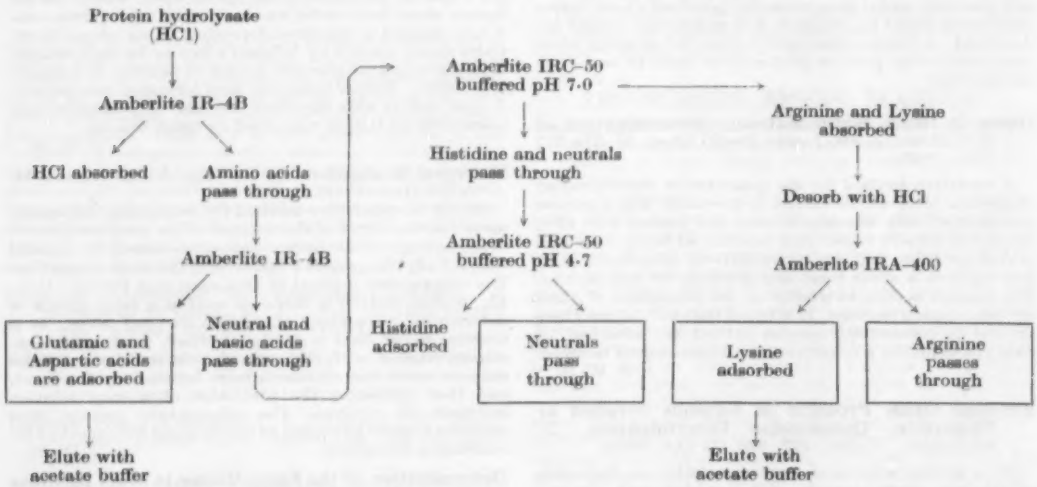
The construction and operation of a manual polarograph are described, and there is an account of polarographic theory in general. Existing literature relating to the use of polarographic methods in the paint industry is reviewed (42 references are given), and the following applications are described in detail— estimation of Pb and Co in driers, of Al in metallic finishes, of Cu in copper naphthenate, of Pb in washings from lead chromate production, and of maleic anhydride in phthalic anhydride. Preliminary studies are reported in the determination of Zn and Ti in pigments, and of the "phthalate content" of resins. J. W. D.

Acetylation of Paper for Use in Chromatography. H. S. Burton. *Chem. and Ind.*, 1229–1230 (14 Nov. 1953).

Whatman No. 1 paper in sheet form is impregnated with 10% $HClO_4$ soln., treated for 1½ hr. at 0°C. with a mixture of acetic anhydride (150 parts), and benzene (350 parts) under constant agitation. After washing with water the sheets are blotted and then dried in a current of air. Over-acetylation must be avoided since it leads to weakening of the paper and loss of fibrous structure. Another suitable paper is produced by acetylation for 15 min. using 4% H_2SO_4 as catalyst. J. W. D.

Ion Exchange and its Applications in the Fine Chemical Industry. E. L. Streatfield. *Chem. and Ind.*, 1214–1222 (14 Nov. 1953).

Amongst the applications which are discussed at length, are water softening, and the resolution of protein hydrolysates. The following scheme for the separation of amino acid mixtures is given; it is due to Winters and Kunin (*Ind. Eng. Chem.*, **41**, 460 (1949))—



Other work in this field is reviewed. J. W. D.

Quantitative Analysis of Mixtures of Mono- and Di-saccharides. W. M. Corbett. *Chem. and Ind.*, 1285 (28 Nov. 1953).

The mono- and di-saccharide contents of mixtures produced by the Lobry de Bruyn–van Ekenstein transformation and by alk. degradation are determined by adaptation of the method of Whistler and Durso (*J. Amer. Chem., Soc.*, **72**, 677 (1950)). The procedure, in which the adsorbate on a charcoal–Celite column is fractionally eluted with water, is described fully. Details are given of the application of the method to eight standard mono–di-saccharide mixtures, the experimental data being tabulated. Results indicate that the total mono- and di-saccharides may be determined in presence of each other with a max. error of ±5%; there is good agreement with results obtained by other methods. J. W. D.

Non-absorptive Light Losses in Microspectrophotometry of Biological Material. P. W. Lange. *Svensk Papperstidning*, **56**, 807–817 (15 Nov. 1953).

After a brief review of the problem of non-absorptive light losses in quantitative microspectrophotometry of biological material, four experiments on cellulose fibres esterified with *p*-phenylazobenzoyl chloride are described which show that swelling of the esterified fibre in pyridine reduces the non-absorptive light losses to about 5%. Methods of preparing cross-sections of short fibres and measuring their thickness are given. S. V. S.

Kürschner–Schweizpacher Method of Estimating Lignin. K. Kürschner and T. Schweizpacher. *Faserforsch. und Textiltech.*, **4**, 507–509 (Dec. 1953); *J. Appl. Chem., U.S.S.R.*, **26**, 1186–1189 (Nov. 1953).

Methoxyl estimation by the Kürschner–Schweizpacher method (cf. J.S.D.C., **69**, 522 (Dec. 1953); also *J. Appl. Chem. U.S.S.R.*, **26**, 1176–1185 (Nov. 1953)) applied to substances predominantly carbohydrate in nature and of low methoxyl content gives variable results attributable probably, to formation of carbon monoxide. This may be oxidised in the first distillate, without simultaneous oxidation of methanol, by adding 10 ml. of 0·5% aq. $AgNO_3$ and boiling under reflux for 10 min. The further procedure is as before. A. E. S.

Apparatus for the Laboratory Testing of the Wash and Scrub Resistance of Water Paints. D. Atherton and W. Peace. *J. Oil and Col. Chem. Assocn.*, **37**, 44–47 (Jan. 1954).

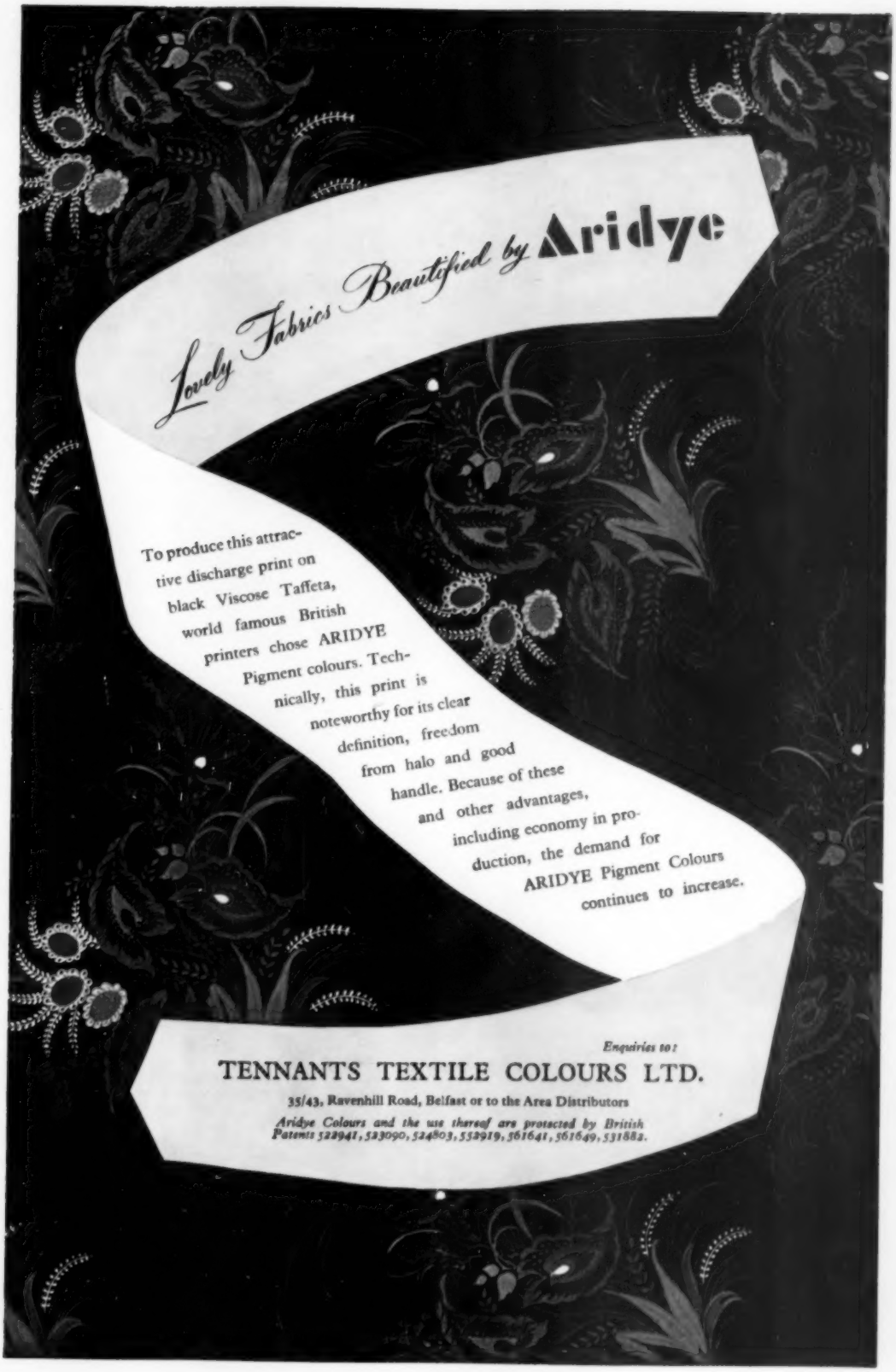
Four paints are tested simultaneously, six tests being carried out on each paint. The surface is rubbed at the rate of 50 6″-strokes per min. with a 1 in. diameter nylon-covered head bearing a 2 kg. load, the paint being kept soaked with 2% Lissapol N soln. Paints showing no failure after 1 hr. (3000 rubs) are judged excellent for wash and scrub resistance and for adhesion to the surface under test. There are three diagrams of the apparatus. J. W. D.

Cadmium Pigments (IV p. 162).
Fractionation of Synthetic Polymers for Fibre Manufacture (VI p. 165).
Detergency and Detergent Testing (VII p. 167).

XV—MISCELLANEOUS

A Pedigree for Three Types of Colour-vision Defect. R. W. Pickford. *Nature*, **172**, 1010 (28 Nov. 1953).

A pedigree is given which is of interest because it includes three types of red–green defect together, these being protanopia, extreme protanomaly, and protanomaly with darkened red; the third is dominant to the second. All the defectives failed the Ishihara test but were not distinguished from one another by it. J. W. B.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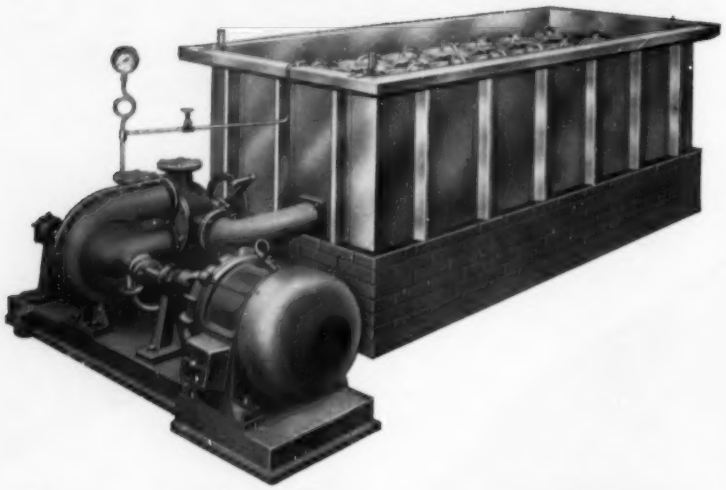

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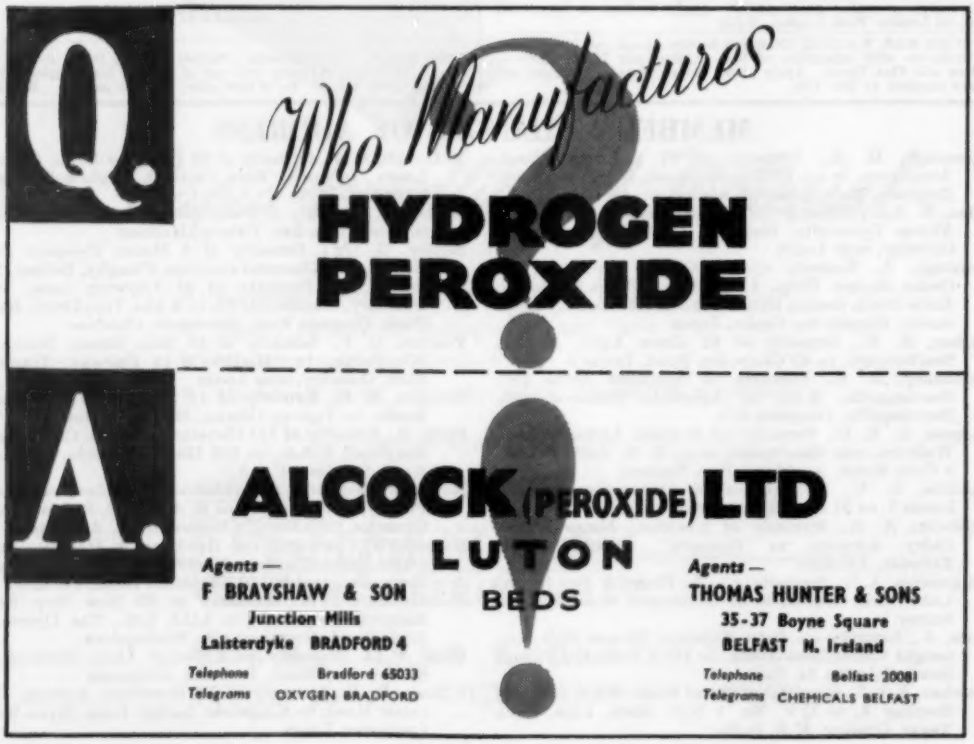

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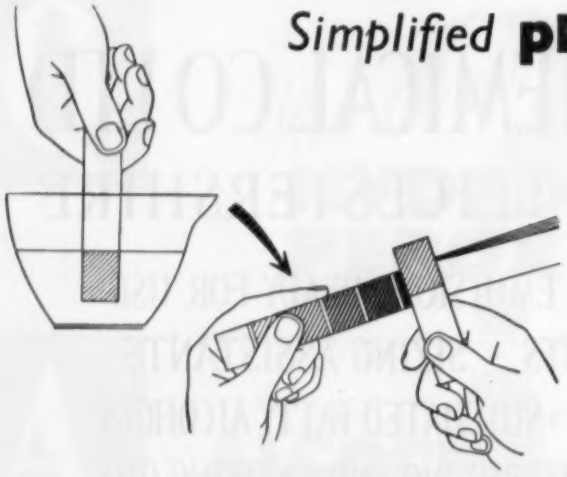

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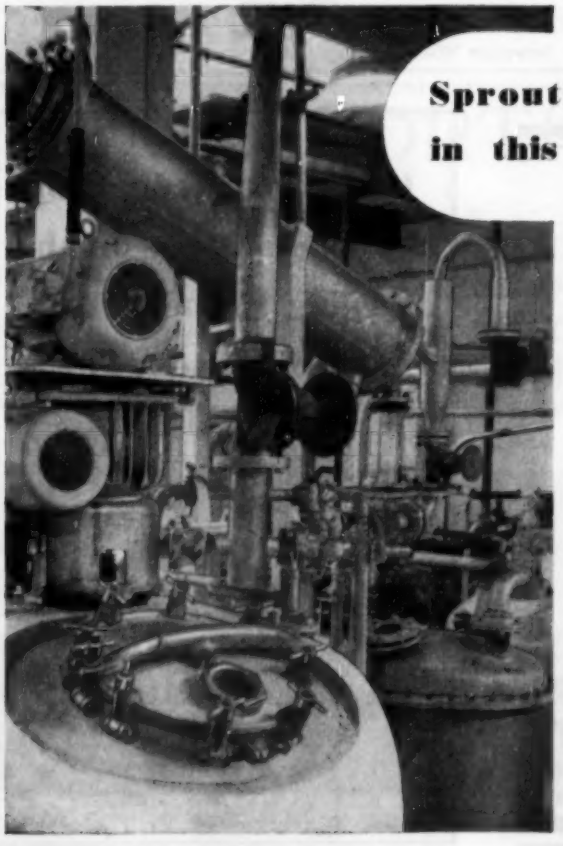

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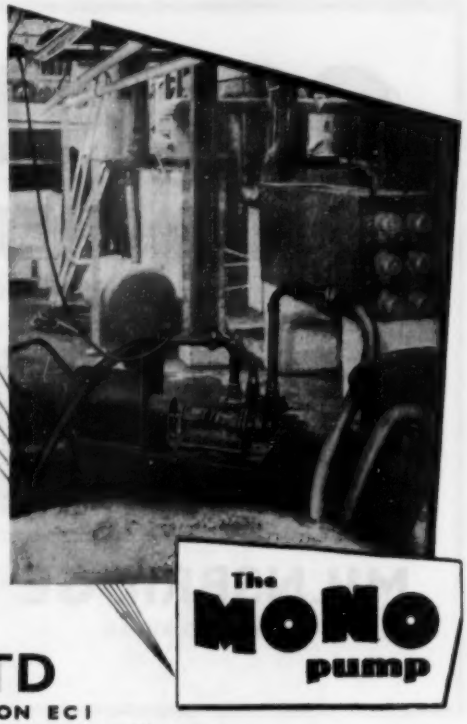

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